Tappi

E JOURNAL OF THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

OCTOBER 1960

VOLUME 43 • NUMBER 10

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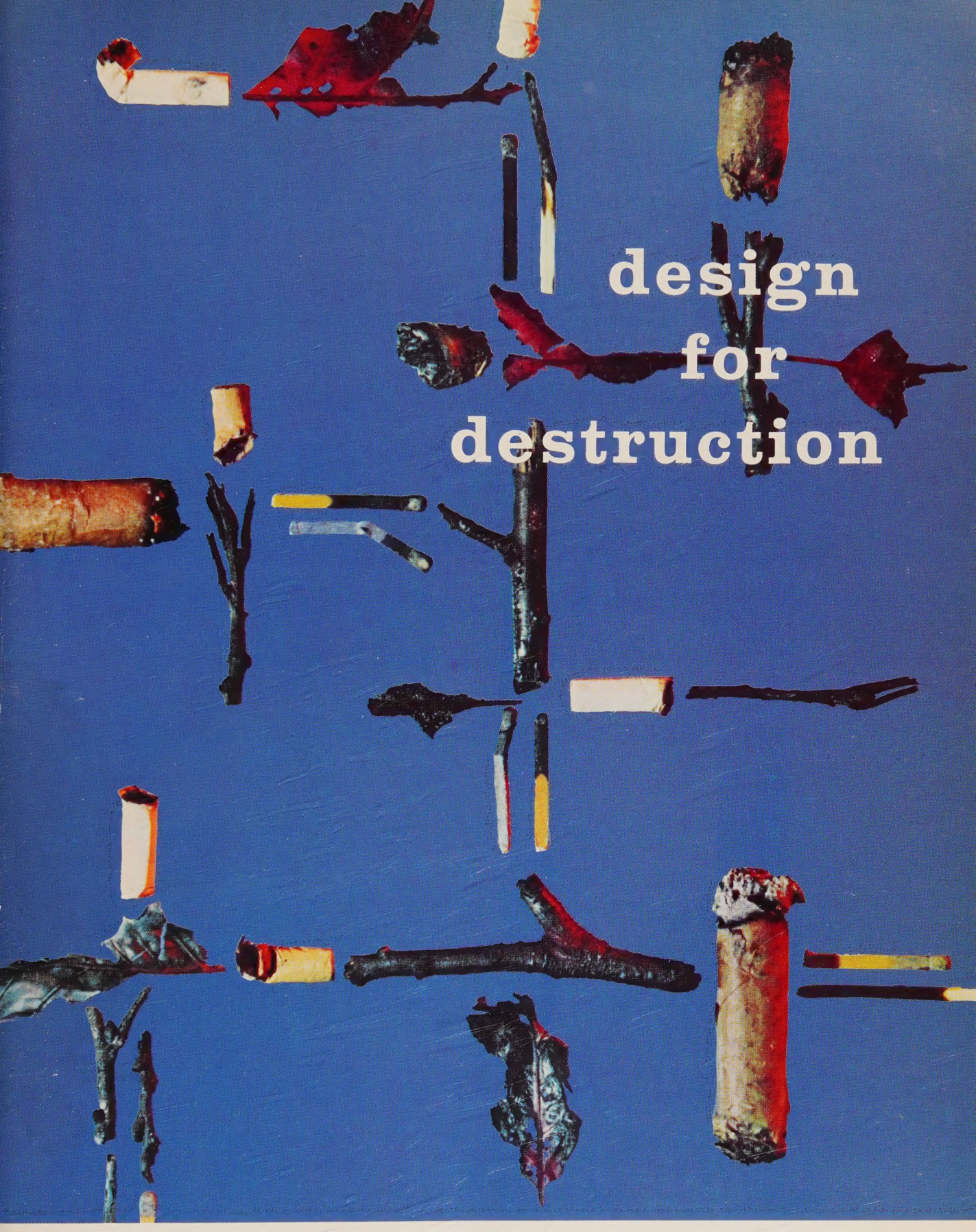

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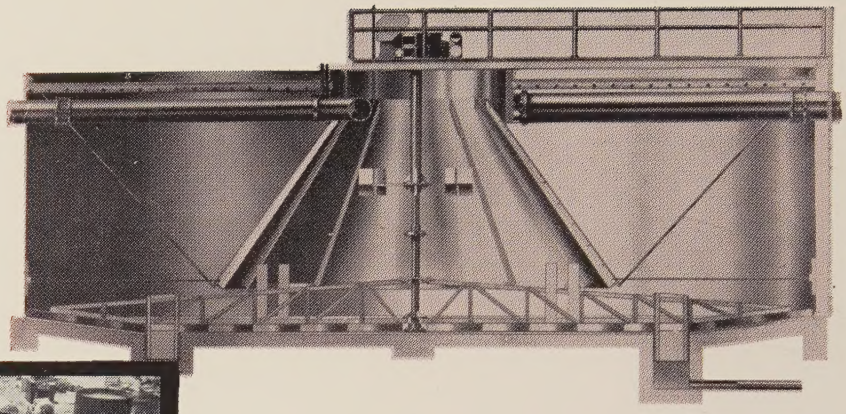

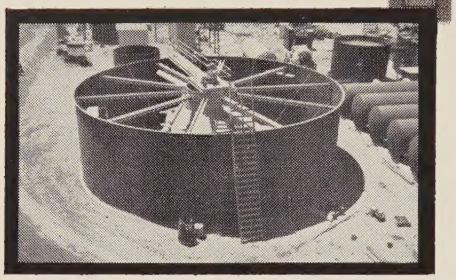

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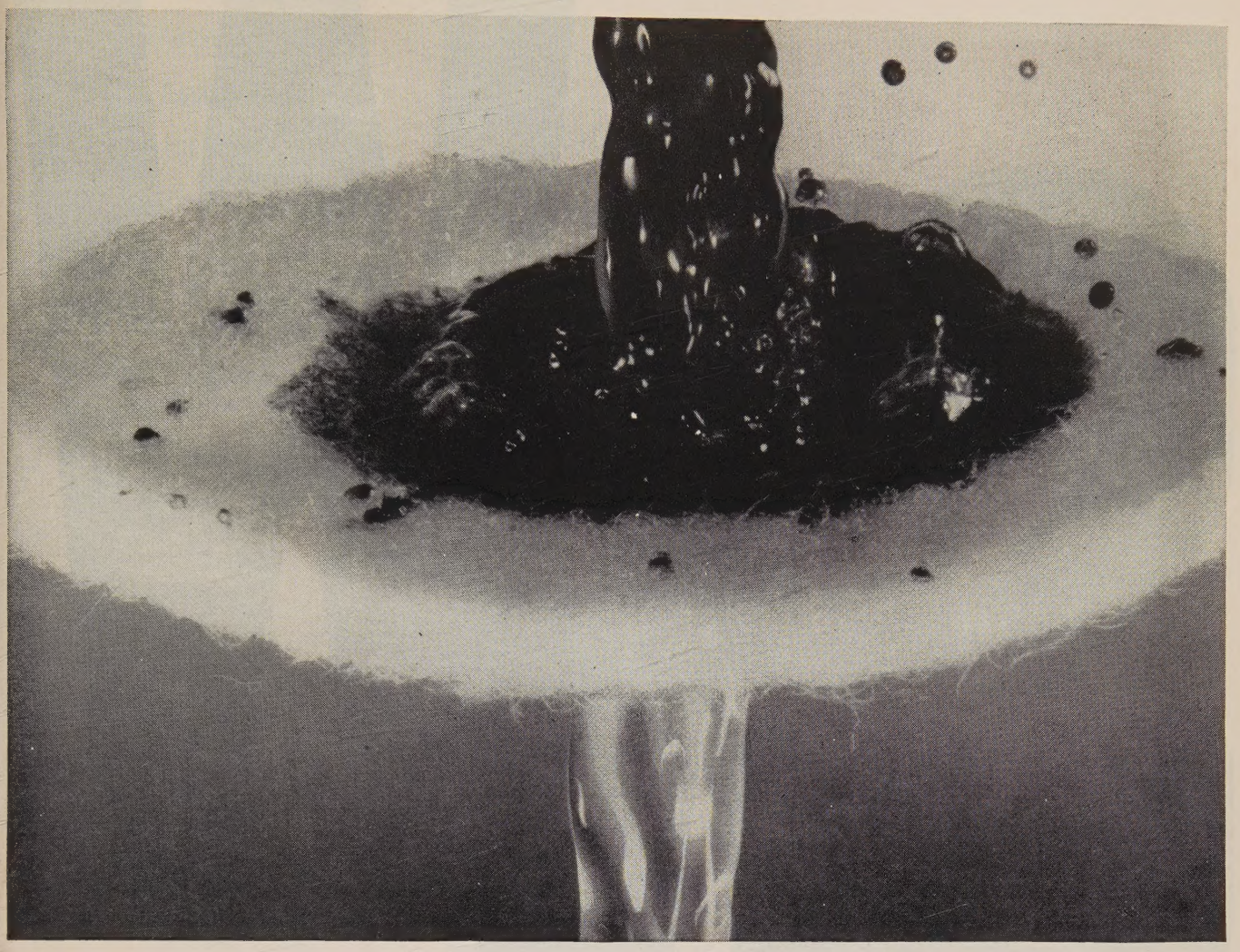

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Calendar of National Conferences

Fifteenth Engineering Conference
Robert Meyer Hotel, Jacksonville, Fla.
Oct. 24–28, 1960

46th Annual Meeting
Commodore Hotel, New York, N. Y.
Feb. 19–23, 1961

Twelfth Coating Conference
Statler-Hilton Hotel, Buffalo, N. Y.
May 22–24, 1961

Twelfth Testing Conference
Queen Elizabeth Hotel, Montreal, Que.
Aug. 15–18, 1961

Eleventh Corrugated Containers Conference
St. Francis Hotel, San Francisco, Calif.
Sept. 11–13, 1961

Please refer to Local Section Activities for the locations and dates of Section meetings

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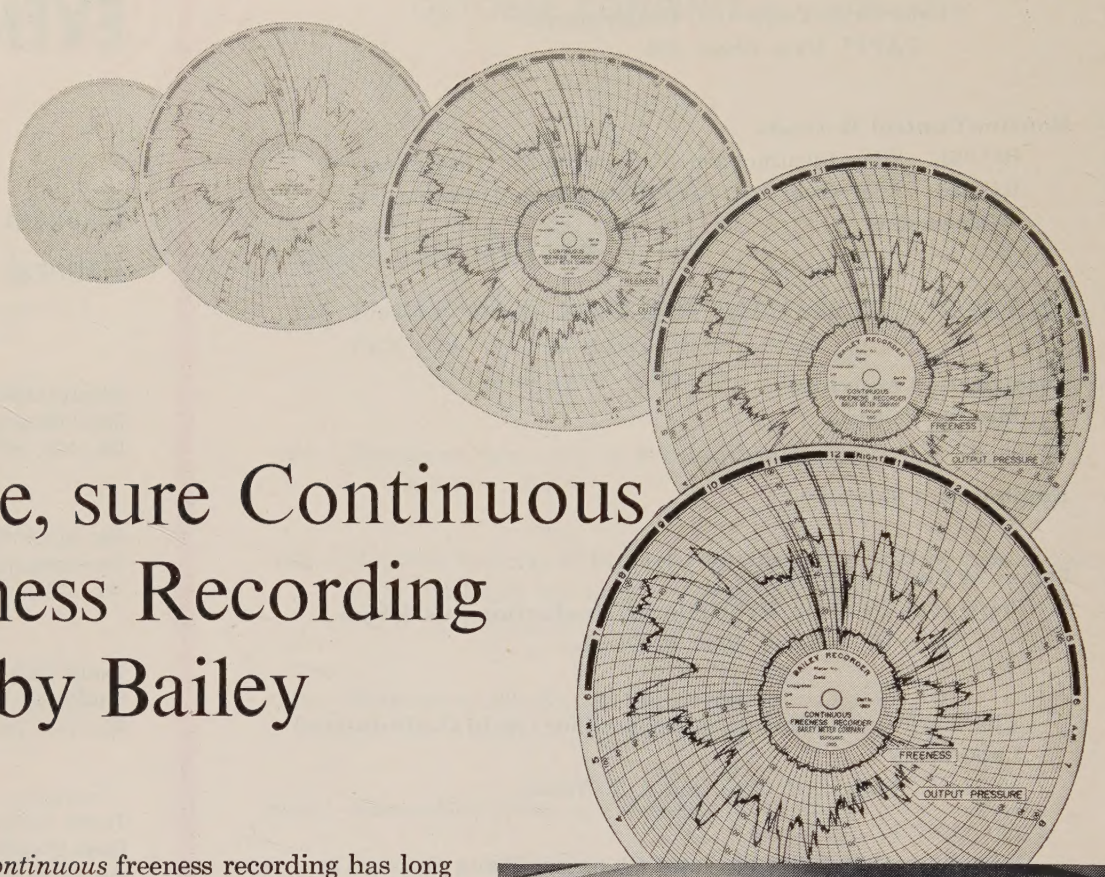

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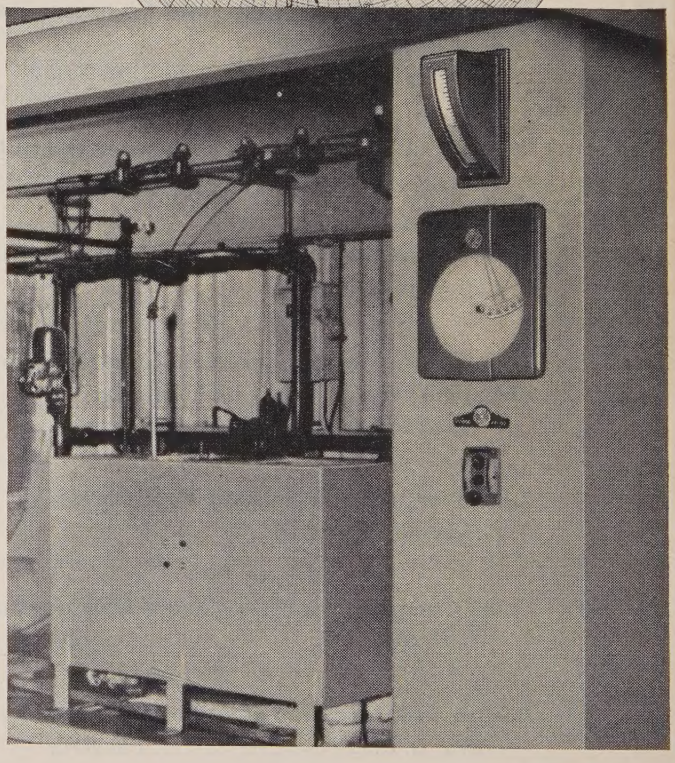

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YELLOW							
Auramine	•	•		•			
Brilliant Pure Yellow	•				•		
Chinoline Yellow	•	•	•		•		
Chrome Yellow	•		•				•
Chrysophenine	•					•	
Direct Yellows	•		•			•	
Fast Light Yellow		•	•		•		
Fastusol® Yellows	•		•			•	
Hansa® Yellow	•		•				•
Metanil Yellow	•	•			•		
Oxide Yellow	•		•				•
Paper Yellow	•					•	
Permagen® Yellow	•		•				•
Stilbene Yellows	•					•	
Tartrazine		•			•		
ORANGE							
Acid Orange	•	•	•		•		
Benzo Fast Oranges	•		•			•	
Chrysoidines	•			•			
Direct Fast Oranges	•		•			•	
Fast Light Orange		•			•		
Fastusol Oranges	•		•			•	
Orange	•	•	•		•		
Permanent Orange	•		•				•
Paper Orange	•					•	
BROWN							
Benzo Brown	•					•	
Bismarck Browns	•		•	•			
Fastusol Browns	•		•			•	
RED							
Benzo Bordeaux	•					•	
Benzo Fast Scarlets	•		•			•	
Benzo Rhoduline® Red	•		•			•	
Brilliant Croceine	•	•	•		•		
Direct Pink	•					•	
Direct Reds	•					•	
Fast Red	•				•		
Fastusol Pink	•		•			•	
Fastusol Reds	•		•			•	
Fastusol Scarlet	•		•			•	
Magenta	•	•	•	•			
Oxide Red	•		•				•
Paper Red	•				•		
Paper Scarlets	•					•	
Permagen Red	•		•				•
Permanent Carmine	•		•				•
Rhodamines	•	•	•	•			
Safranine	•	•	•	•			
Solar® Pink	•		•				•
Solcarbo® Pink	•		•			•	

	APPLICATION: Beater	Calender	Coating	CLASS: Basic	Acid	Direct	Pigment
VIOLET							
Brilliant Benzo Violet	•					•	
Crystal Violet	•	•		•			
Ethyl Violet	•	•		•			
Fastusol Violets	•					•	
Formyl Violet	•	•			•		
Methyl Violet	•	•		•			
Solar Coating Violet			•				•
Solar Violet	•		•				•
BLUE							
Benzo Azurine	•		•			•	
Benzo Blues	•		•			•	
Benzo Sky Blue	•		•			•	
Brilliant Benzo Blue	•		•			•	
Brilliant Wool Blue		•			•		
Fast Bond® Blues	•				•		
Fastusol Blue	•		•			•	
Fastusol Turquoise Blue	•		•			•	
Heliogen® Blues	•		•				•
Methylene Blue	•	•	•	•			
Neptune® Blues		•	•		•		
Paper Pure Blue	•	•	•	•			
Rhoduline Blues	•	•		•			
Solar Blues	•		•				•
Solar Coating Blue			•				•
Solcarbo Blues	•		•			•	
Soluble Blues	•				•		•
Victoria Blue	•	•	•	•			
Victoria Pure Blue	•	•	•	•			
GREEN							
Benzo Greens	•					•	
Brilliant Green	•	•	•	•			
Fast Bond Green	•				•		
Fastusol Greens	•					•	
Guinea Green	•	•	•		•		
Heliogen Greens	•		•				•
Heliogen Viridines	•		•				•
Naphthol Green		•	•		•		
Neptune Green	•	•	•		•		
Pigment Green	•		•				•
Solar Green	•		•				•
Victoria Green	•	•	•	•			
BLACK							
Acid Black		•			•		
Basic Black	•	•		•			
Diamine Black	•		•			•	
Diazo Black	•		•			•	
Direct Deep Black	•		•			•	
Fastusol Greys	•		•			•	
Naphthylamine Black	•	•	•		•		
Nigrosines	•		•			•	
Permanent Black	•		○				•

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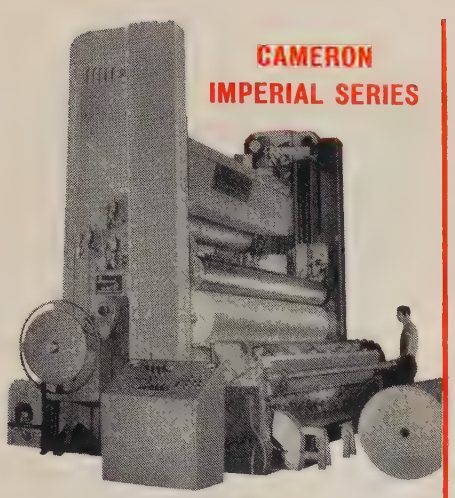

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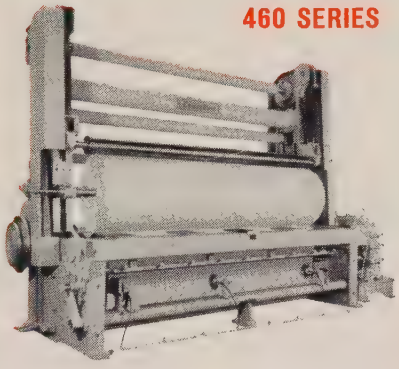

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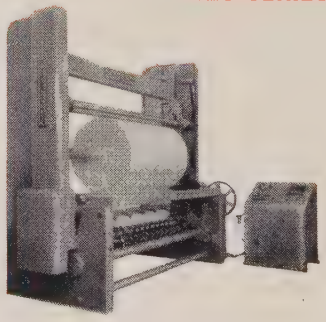

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Relative Toxicity of Biostatic Agents Suggested for Use in the Pulp and Paper Industry 1958 Review

J. H. CONKEY and J. A. CARLSON

THE Biological Control Committee of the American Paper and Pulp Association sponsors at The Institute of Paper Chemistry, as a part of its general program of biological research, a laboratory investigation of the relative toxicity of various biostatic materials offered to the pulp and paper industry. This program was initiated in 1941 (*2*) for the purpose of providing the industry with comparative toxicity data on commercially available slime-control agents. The program has since been extended to cover materials suggested for uses other than slime control and certain experimental materials believed to be of potential value.

In 1953 the Biological Control Committee published a complete review (*6*) of all prior data obtained under this program (*3, 4, 5*). Also, a policy was established, whereby, such a review would be published at five-year intervals with current data supplied yearly in the form of supplements (*7–10*). A five-year plateau has now been reached and, therefore, this report includes a tabulation of all relative toxicity data obtained to date in addition to the work completed during the past year. The total number of compounds thus far screened 192. Further breakdown shows that 91 of these compounds were tested prior to the 1953 review, an additional 79 were covered during the years 1954 through 1957, and 22 are materials tested during the past year.

EXPERIMENTAL PROCEDURE

The test method used, basically an agar-Petri plate technique, evolved from a method suggested by Schmitz, *et al.* (*1*), for the testing of wood preservatives. The method has undergone a number of modifications since the start of this program. Recently, changes in technique have been made to shorten testing time and certain media changes also have become necessary. It was considered desirable, therefore, to include in this report a description of the test method as it is now practiced.

The inhibiting concentration of the test materials was determined for two species of bacteria (*Aerobacter aerogenes, Bacillus mycoides*) and two species of fungi (*Aspergillus niger, Penicillium expansum*). The bacterium *Aerobacter aerogenes* is a nonspore-forming organism. It is capable of forming capsules under certain conditions and is frequently isolated from pulp and paper mill systems. The particular strain of *A. aerogenes* used was obtained from the University of California. The organism *Bacillus mycoides* is a spore-forming organism also frequently isolated from pulp and paper mill systems. The *B. mycoides* strain used was originally isolated from a sample of paperboard. The fungus species *Aspergillus niger* and *Penicillium expansum* are common to pulp and paper mill systems, stored pulps, and finished paper products. The presently used strain of *A. niger* was obtained from Buckman Laboratories (BL89) and the source of the culture *P. expansum* was the American Type Culture Collection (ATCC 1117).

As the test substrate two types of nutrient media are required. For the cultivation and testing of the bacterial species Bacto*-Tryptone Glucose Extract Agar (TGE) has been substituted for the Bacto-Nutrient Agar used previously due to the difficulty experienced in obtaining inhibition of the bacterial species at the control concentrations of phenylmercuric acetate. This difficulty was traced to a phenylmercuric acetate inactivating factor present in the medium which apparently varied between different purchase lots of the medium. A number of lots of TGE Agar were checked and found to be more satisfactory than Nutrient Agar in obtaining inhibition of the bacterial species at the desired control levels. Rechecks made of materials representative of each of the general groups of compounds (mercurials, chlorinated phenols, etc.) using TGE Agar, however, showed no significant differences in inhibitory levels when the controls were at normal levels (Table I).

A change has also been made in the medium used in testing the effect of the various compounds toward the fungi. Previously a malt agar was used which consisted of 15 g. of Plain Trommers Diastasic Extract of Malt and 20 g. Bacto-Agar per liter of distilled water; however, the Trommers malt is no longer available commercially. Experiments with various media (Tables II and III) led to the selection of a medium consisting of 15 g. Bacto-Malt Extract and 15 g. Bacto-Agar per liter of distilled water as a replacement for the previous medium.

The medium was prepared on the day of a test run, dispensed into 150-ml. dilution bottles, autoclaved at 15 p.s.i. for 20 min., and stored at 50°C. until time of use. Formerly, the exact quantity of medium per bottle was determined by weighing the bottles prior to filling and reweighing after filling and sterilization. A considerable saving in time has resulted by pipetting*

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Table I. Performance of Representative Materials in Various Media

Biostatic agent	Inhibiting concentration, p.p.m. — Aerobacter aerogenes, Nutrient agar	Aerobacter aerogenes, TGE agar	Bacillus mycoides, Nutrient agar	Bacillus mycoides, TGE agar
Butyl Parasept	150	150	80	80
Dowicide B	20	30	15	25
Dowicide G	200	250	4	5
Gallosan	50	60	7	5
8-Hydroxyquinoline	70	80	3	3
Lignasan	10	10	2	3
Phenylmercuric acetate	2	2	0.2	0.2

Table II. Performance of Phenylmercuric Acetate in Various Media

Medium	Composition	Inhibiting concentration, p.p.m. — Aspergillus niger	Penicillium expansum
A	15 g. Trommers diastasic extract of malt 20 g. Bacto-agar 1 l. Distilled water	5	3
B	15 g. Bacto-malt extract 15 g. Bacto-agar 1 l. Distilled water	5	3
C	39 g. Bacto-potato dextrose agar 1 l. Distilled water	5	3
D[a]	45 g. Bacto-malt agar 1 l. Distilled water	7	3
E[a]	33.6 g. Bacto-malt extract agar 1 l. Distilled water	5	3
F[a]	15 g. BBL malt extract 15 g. Bacto-agar 1 l. Distilled water	3	3

[a] At levels where growth took place the extent and appearance varied markedly from medium A.

Table III. Performance of Representative Materials in Various Media

Biostatic agent	Inhibiting concentration, p.p.m. — Aspergillus niger, Medium A[a]	Aspergillus niger, Medium B[a]	Aspergillus niger, Medium C[a]	Penicillium expansum, Medium A[a]	Penicillium expansum, Medium B[a]	Penicillium expansum, Medium C[a]
Dowicide G	30	30	35	35	35	35
Gallosan	90	90	90	45	45	55
8-Hydroxyquinoline	100	100	80	35	35	25
Lignasan	40	50	30	25	25	15
Phenylmercuric acetate	5	5	4	4	4	4
Propyl Parasept	450	450	450	350	350	350

[a] See Table II.

Table IV. Toxicant Increments

Range, p.p.m.	Increment, p.p.m.
0.01–0.1	0.01
0.1–1.0	0.1
1.0–10	1.0
10–100	5.0
100–1000	50.0
1000–3000	500.0

a known quantity of medium into each bottle. The weighing procedure was followed in initial runs to determine the delivered weight after sterilization and the coefficient of variation found was approximately ±0.5%. Duplicate plates are poured from each bottle.

The test materials were freshly prepared as stock solutions using distilled water as the solvent whenever possible. Ethanol (95%) and acetone were used if necessary; however, the concentration of organic solvent in the medium was not allowed to exceed 2.5%.

The stock solutions were prepared on a weight basis only and the volumes added to the substrate maintained in the range of 0.5 to 5.0 ml. Additions were calculated according to the following formula:

$$\text{Ml. addition} = \frac{\text{Desired concn.} \times \text{medium wt.}}{\text{Concn. of stock soln.} - \text{desired concn.}}$$

The concentration increments used are presented in Table IV. A control series using a purified grade of phenylmercuric acetate was included in each test run as a check on the sensitivity of the test organisms. Normal inhibition levels are: *Aerobacter aerogenes*, 2.0 p.p.m.; *Bacillus mycoides*, 0.2 p.p.m.; *Penicillium expansum*, 6.0 p.p.m.; and *Aspergillus niger*, 5.0 p.p.m.

The bacterial inoculum was set up by transferring the organism from a 24-hr. test tube slant to a bottle slant. (The bottle slants were prepared by slanting 25 ml. of TGE Agar in an 8-oz. square pyrex dilution bottle.) The *A. aerogenes* bottle slants were incubated for 24 hours while the slants of the spore-forming organism *B. mycoides* were incubated for 72 hr. To prepare the inoculum the entire growth from the surface of the bottle slant was suspended in 100 ml. of distilled water and two 3-mm. loopfuls used to inoculate each poured plate. The plates were read after 48 hr. incubation at 37°C. and the concentration level at which all growth ceased was considered as the point of inhibition.

The fungus inoculum was obtained by cutting disks 6 mm. in diameter from the periphery of a two-week old culture using a cork borer. It has been found helpful in the cutting and handling of the fungus disks to make the inoculum medium firmer by increasing the agar content to 3%. A disk of inoculum was placed in the center of each test plate with the fungus mat in contact with the test substrate. The fungi plates were incubated at 28°C. and final observations made after 14 days' incubation. As in the case of the bacteria, the concentration level showing no growth on the test substrate is considered as the point of inhibition.

Table V. Materials Tested in 1957

Biostatic agent	Inhibiting concentration, p.p.m. — Aerobacter aerogenes	Bacillus mycoides	Aspergillus niger	Penicillium expansum
Bio 2273	>3000	40	95	100
Bio 2274	>3000	70	200	300
Bio 4174	>3000	150	>3000	2500
3-Bromothianaphthene-1-dioxide	2000	65	3000	55
Chemocide MSS	200	6	2000	750
2-Chlorothianaphthene-1-dioxide	150	45	65	45
3-Chlorothianaphthene-1-dioxide	400	50	200	65
DAC 559	250	150	550	300
2,3-Dibromothianaphthene-1-dioxide	850	15	3000	2000
2,3-Dichloro-2,3-dihydrothianaphthene-1-dioxide	150	40	85	60
Fungitrol 50	45	20	>3000	1500
MetaSalt P	2	0.1	4	3
Meta-san E 100	3	0.6	3	2
Meta-san Krylate	7	0.5	6	3
Meta-san M 100	4	0.6	4	2
Meta-san R	9	0.7	20	8
Nuosept	500	40	500	400
Nuozene	>3000	150	75	>3000
SCXP 1170	90	20	70	35
SCXP 1290	95	25	90	55
Zonemask	>3000	50	>3000	>3000
Zonemask L-39	>3000	20	>3000	>3000
Phenylmercuric acetate control	2	0.2	5	6

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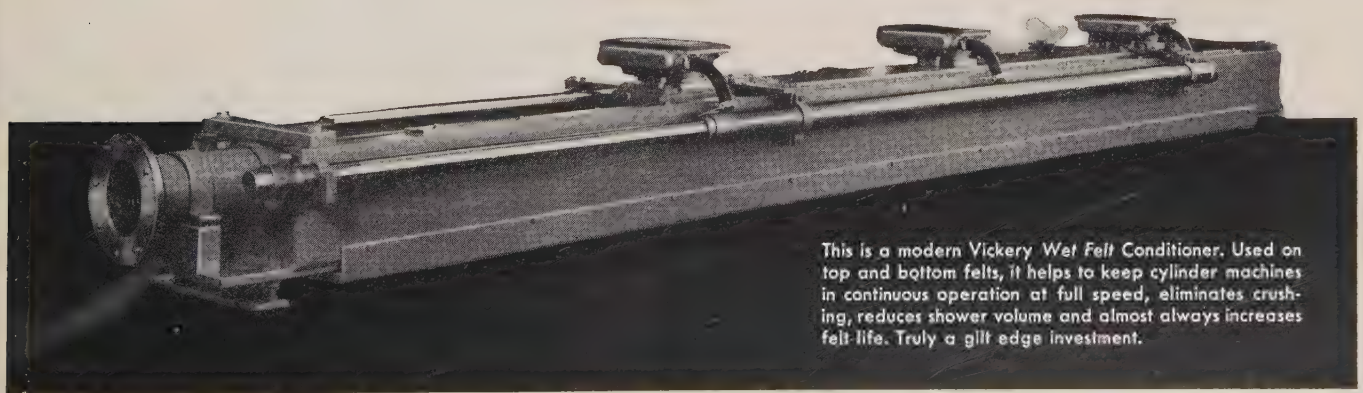

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Table VI. Data on All Materials Tested

Biostatic agent	Inhibiting concentration, p.p.m. Aerobacter aerogenes	Bacillus mycoides	Aspergillus niger	Penicillium expansum
A-36	20	4	45	35
ATM-50	500	4	>3000	2500
ATM-X3	850	9	>3000	2000
5-Acetoxymercurivanillin	5	0.7	2000	40
Actamer	30	0.2	>3000	>3000
Advacide PMN	9	1	25	35
Advacide PMO	15	0.8	30	30
Aminoethyl ethanolamine	2000	2500	>3000	>3000
Armac CD 50	150	65	>3000	>3000
Arquad 2C	1500	35	>3000	>3000
Arquad 2C Oleate	>3000	40	>3000	>3000
Arquad 12	80	65	>3000	>3000
Arquad 16	450	5	>3000	>3000
Arquad 18	>3000	55	>3000	>3000
Arquad S	1500	35	>3000	>3000
Arquad S-2C	650	6	>3000	>3000
BCD 955	850	40	2000	1000
Becco peracetic acid 40%	300	250	1500	950
Benochlor 3C	>3000	75	>3000	>3000
Benzyl Parasept	85	45	>3000	>3000
Bio 2273	>3000	40	95	100
Bio 2274	>3000	70	200	300
Bio 4174	>3000	150	>3000	2500
Bionol A	150	15	>3000	>3000
Bis-(2-chlorethyl)-formal	>3000	2500	>3000	2500
3-Bromothianaphthene-1-dioxide	2000	65	>3000	55
BSM-11	25	3	25	25
BSM-11B	15	3	45	55
Buffen 30	6	0.8	20	30
Busan	400	4	750	400
Butrol	20	2	45	30
Butrol 8	4	0.4	20	20
Butyl Parasept	150	80	>3000	250
Cadminate	300	60	2000	500
Calcium formate	>3000	>3000	>3000	>3000
Caso-cide (Permacide AM 10)	40[a]	15[a]	>3000	800
Cetylpyridinium bromide	550	7	>3000	>3000
Chemocide MMS	200	6	2000	750
Chloroben	>3000	70	>3000	>3000
Chloro-isophorone	250	20	250	150
2-Chloromercurithiophene	3	0.2	30	15
4-Chlororesorcin	700	500	2500	1500
2-Chlorothianaphthene-1-dioxide	150	45	65	45
3-Chlorothianaphthene-1-dioxide	400	50	200	65
Copper pentachlorophtenate	350	3	25	45
Copper 3-phenylsalicylate	550	150	450	400
Cunilate 2493	...	...	450	2000
Cunimene D 2536	...	...	550	700
Cunimene D 2601	...	...	150	150
Cunimene D 2607	...	...	150	150
DAC 559	250	150	550	300
DHAS	>3000	3000	450	350
2,3-Dibromothianaphthene-1-dioxide	850	15	3000	2000
2,3-Dichloro-2,3-dihydrothianaphthene-1-dioxide	150	40	85	60
Diphenylguanidium trichloroacetate	700	550	3000	2000
Dowicide A	200	200	150	150
Dowicide B	20	15	15	7
Dowicide C	60	30	55	30
Dowicide D	45	20	65	50
Dowicide E	60	15	150	80
Dowicide F	400	7	20	30
Dowicide G	200	4	25	30
Dowicide H	200	8	20	30
Dowicide K5	250	4	600	350
Dowicide S	85	15	30	45
Dowicide 2S	200	40	20	15
Dowicide 31	40	25	35	35
Drew liquid slime control	10	2	30	35
Drew powder	2	0.3	6	4
Duomeen C	70	55	3000	700
Duomeen CDA 50	250	80	>3000	>3000
Dynacide	15	3	65	65
Emulsept E607 special	400[a]	150[a]	>3000	>3000
Ethyl formate	>3000	400	>3000	>3000
Ethylmercuric methacrylate	20	5	20	20
Ethyl Parasept	700	600	600	450
Experimental slimicide no. 5	150	350	300	1500
Formaldehyde-*p*-nitrophenylhydrazone	250	75	>3000	400
Fungicide 974 (CRAG)	50	20	85	45
Fungitrol 50	45	20	>3000	1500
G4	25	2	200	200
Germycin-5	7	0.5	25	20
Gallicide	20	4	150	150
Gallosan	50	7	90	45
Hydrazine difuroate	1000	80	3000	3000
5-Hydroxymercurivanillin	7	0.6	>500	45
8-Hydroxyquinoline	70	3	100	45
Lignasan	10	2	40	15
Merfenel	1	0.1	4	4
Merfenel A (20% solution)	15	0.7	25	25
Merfenel 51	9	0.5	35	25
Mersynate	20	6	20	20
MetaSalt P	2	0.1	4	3
Meta-san 17	3	0.3	6	7
Meta-san 35	3	0.3	6	7
Meta-san E 100	3	0.6	3	2
Meta-san Krylate	7	0.5	6	3
Meta-san M 100	4	0.6	4	2
Meta-san P	2	0.2	5	5
Meta-san PN	5	0.3	8	8
Meta-san POS	30	3	40	30
Meta-san R	9	0.7	20	8
Meta-san W	15	0.9	15	6
Metasol A	15	0.9	45	55
Metasol Krylate G	40	3	30	30
Methylmercuric (2,4-dichlorophenoxy) acetate	25	7	25	20
Methylmercuric hydroxide	35	7	25	25
Methyl Parasept	1500	800	850	550
Microdyn	2000	2000	>3000	>3000
Monobutyl *m*-cresol	150	50	300	400
Movidyn	300	2	>3000	>3000
NC 1235	65	3	>3000	2500
NC 2050	7	0.7	25	5
Negant 30 s	8	0.5	25	30
NP 1711	75	55	95	75
NP 1713	450	40	20	25
Nacconol FSNO	>2000	20	1500	...
Nalco 21	750	8	2000	950
Nalco 21B	200	25	55	550
Nalco 21M	200	5	45	40
Nalco 21S	25	5	35	55
Nalco 23	450	10	35	70
Nalco 234	>3000	25	>3000	>3000
No Slyme	55	5	80	100
Nuodex copper NOH-6%	800	35	>3000	>3000
Nuosept	500	40	500	400
Nuozene	>3000	150	75	>3000
Nytron	>3000	150	>3000	>3000
Ottacide	80	20	30	50
Ottafect	35	6	95	75
Ottasept 430	80	20	30	50
P1006	>3000	>3000	>3000	>3000
P200A	35	5	50	55
P.M. 32 Powder	15	2	60	55
PMA	3	0.4	7	9
PMAS	9	0.7	45	45
Penetox	500	30	>3000	>3000
Penn Salt C.A.F.	2000	2500	2000	2500
Perma Cide 135	50	50	>3000	>3000
Perma Cide AL-10	25	3	95	150

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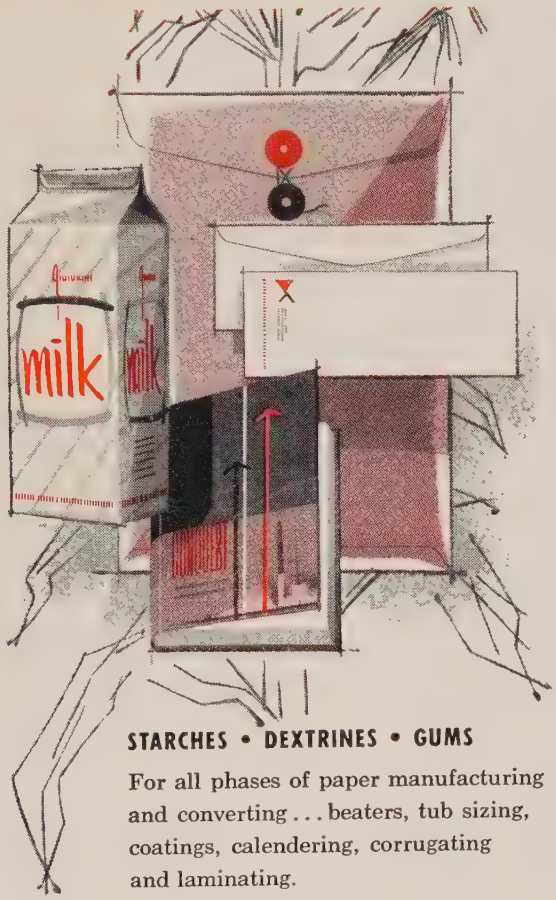

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Biostatic agent	*Inhibiting concentration, p.p.m.* Aerobacter aerogenes	Bacillus mycoides	Aspergillus niger	Penicillium expansum
Permatox 1S	35	2	35	30
Permycin-5	30	6	350	100
Permycin C-5	35	8	450	95
Phenylmercuric undecylinate	35	5	45	45
Phymer	50	20	>3000	>3000
Polymyxin B sulfate	40	950	>1000	>1000
Preventol GD	20	2	150	150
Preventol GDC	45	5	300	500
Propargyl-*N*-phenyl carbamate	550	500	300	200
Propyl Parasept	250	250	450	350
Purasan AD	65	6	>3000	>3000
Purasan DC	700	3	>3000	>3000
Purasan X-3	550	4	>3000	>3000
Puratized 300	25	15	2500	>3000
Puratized 641	35	2	40	35
Puratized 1122	25	15	600	300
Puratized 1122 (10% emulsifiable solution)	150	80	3000	1500
Puratized F	8	2	25	30
Puratized N5DS	15	1	35	45
Puratized N5E	20	4	65	70
Puratized SC	3	0.6	6	6
Pyridylmercuric acetate	1	0.2	95	25
Pyridylmercuric chloride	1	0.2	95	20
Quicksan EM	15	2	30	45
Ramplex C 106	25[a]	4[a]	40[a]	. . .
Roccal	200[a]	7[a]	>3000	>3000
RD-1652	>3000	250	>3000	>3000
Rosin-Amine D acetate	30	15	>3000	>3000
Santobrite	250	4	35	30
Santophen 1	55	15	80	80
SC 100	10	1	10	70
SC 200	3	0.5	4	5
SCXP	90	20	70	35
SCXP 1290	95	25	90	55
S-ethyl mercury thioammeline	2	0.5	2	5
Slime-Acide L.M.	3	0.5	15	30
Slime-Acide M	1	0.3	2	. . .
Slime-trol RX3	600	9	90	95
Slimicide MSC	60	40	>3000	>3000
Slimodyn	30	2	80	25
Sodemmet	4	0.7	3	3
Sodium salt 2-pyridinethione	3	0.5	65	20
2,4,6-Trichloro-resorcinol	30	15	150	100
Tris-*beta*-chloro-ethyl phosphate	3000	2000	>3000	>3000
Tween 80	>3000	>3000	>3000	>3000
Tyrothricin	>3000	20	>3000	>3000
USF 188	350	200	400	400
Vancide 32	950	400	550	800
Vancide 51 (30% solution)	95	2	2000	750
Vancide F-999	400	4	30	25
Zonemask	>3000	50	>3000	>3000
Zonemask L-39	>3000	20	>3000	>3000
Phenylmercuric acetate control	2	0.2	5	6

[a] Inhibiting concentration based on v/w (3).

Where additional growth appears to occur only on the inoculum disk, the disk is moved to determine whether or not penetration of the test substrate has taken place. Without penetration of the test medium the fungus was rated as inhibited. (Sterile equipment and aseptic techniques were used throughout the test procedure.)

EXPERIMENTAL RESULTS AND DISCUSSION

The data obtained from the work done during the past year and which have not been previously reported are listed in Table V. Table VI presents the complete data on all compounds tested under this program up to the present time. The active ingredients and sup-

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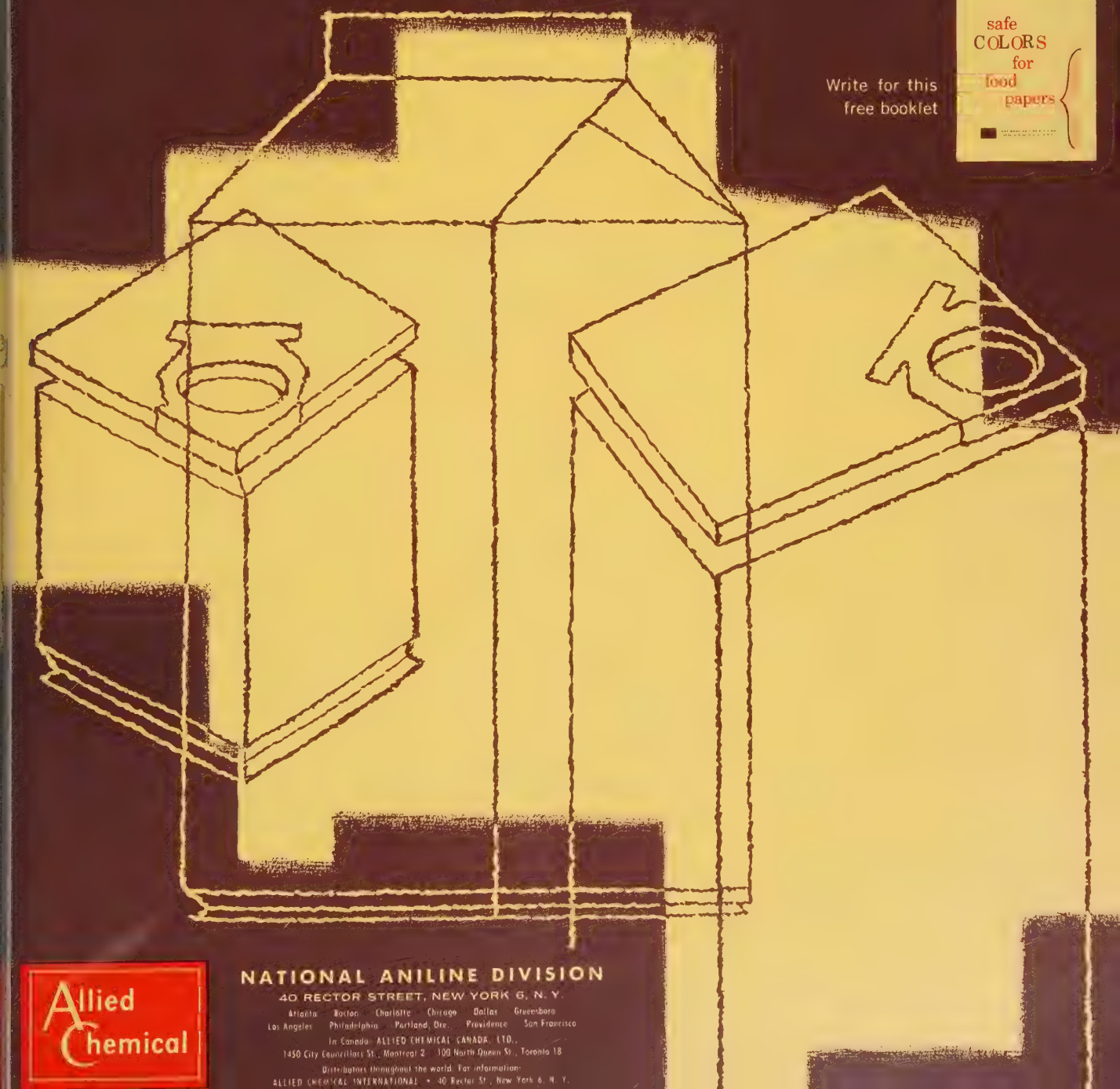

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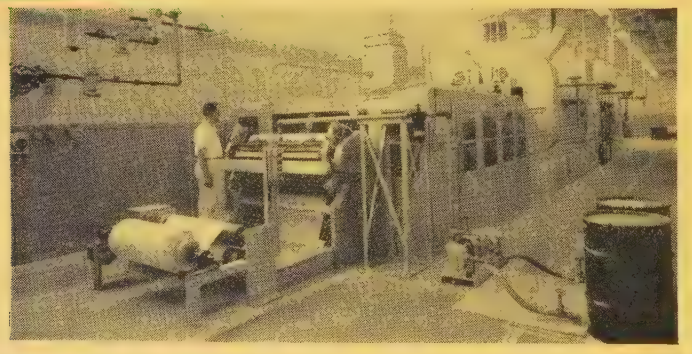
This Waldron Coater is available as a single unit, or as an integral part of completely engineered coating line, as illustrated. In such an integrated line selected standard Waldron Web-Processing Units are used in combination with the Waldron Reverse Roll Coater and with the drying equipment designed by our Ross Division, specialists in air systems.

Our technical bulletin on Reverse Roll Coating gives full details. We'll be glad to send you a copy; also a bulletin describing how companies like yours can use our fully equipped and staffed coating and drying laboratory for test and development purposes.



USERS OF WALDRON REVERSE ROLL COATERS

* AC Spark Plug Company
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Alsco, Inc.
American Decalcomania Co., Inc.
Armstrong Cork Company
Arvey Corporation
* Bauer and Black
Bird and Son, Inc.
Boeing Airplane Company
L. E. Carpenter Company
Chandler Manufacturing Co., Inc.
Columbus Coated Fabrics Corp.
Commercial Plastics, Ltd.
Consolidated Cigar Company
* Crown Cork and Seal Co., Inc.
* Dennison Manufacturing Company
* Di-Noc Chemical Arts, Inc.
Douglas Aircraft Co., Inc.
* E. I. duPont deNemours and Co., Inc.
* Gaylord Container Corporation
General Cigar Company, Inc.
* General Electric Company
Grant Photo Products, Inc.
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* Irvington Varnish and Insulator Co.
* Johnson and Johnson
Keller Dorian Company
* Kyowa Rubber Industry Co., Ltd.
Les Etablissement Marechal
* McLaurin-Jones Company
* Meyercord Corporation
* Mica Insulator Company
Munising Paper Company
Mystik Adhesive Products
North American Aviation, Inc.
Odell Company
Paper Affiliates
* Peerless Photo Products, Inc.
* Permacel
* Plastic Film Corporation
Plymouth Rubber Company
Premoid Corporation
Prescott Textile Manufacturing Co., Inc.
* Racquette River Paper Company
Respro Division of General Tire and Rubber Company
* Riegel Paper Company
Rubber and Asbestos Corporation
A. Sanderson Company
* Sandura Company
Service Backing and Coating Corp.
* Standard Cap and Seal Company
* Standard Coated Products Company
* Strobridge Lithographing Company
* Synthane Corporation
Textileather Corporation
Tobacco Nuforms, Inc.
* U. S. Rubber Company
U. S. Steel Corporation
* Visking Company Division of Union Carbide Canada, Ltd.
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* *Completely integrated systems in combination with Ross Dryers*

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RESIN & RUBBER LATICES

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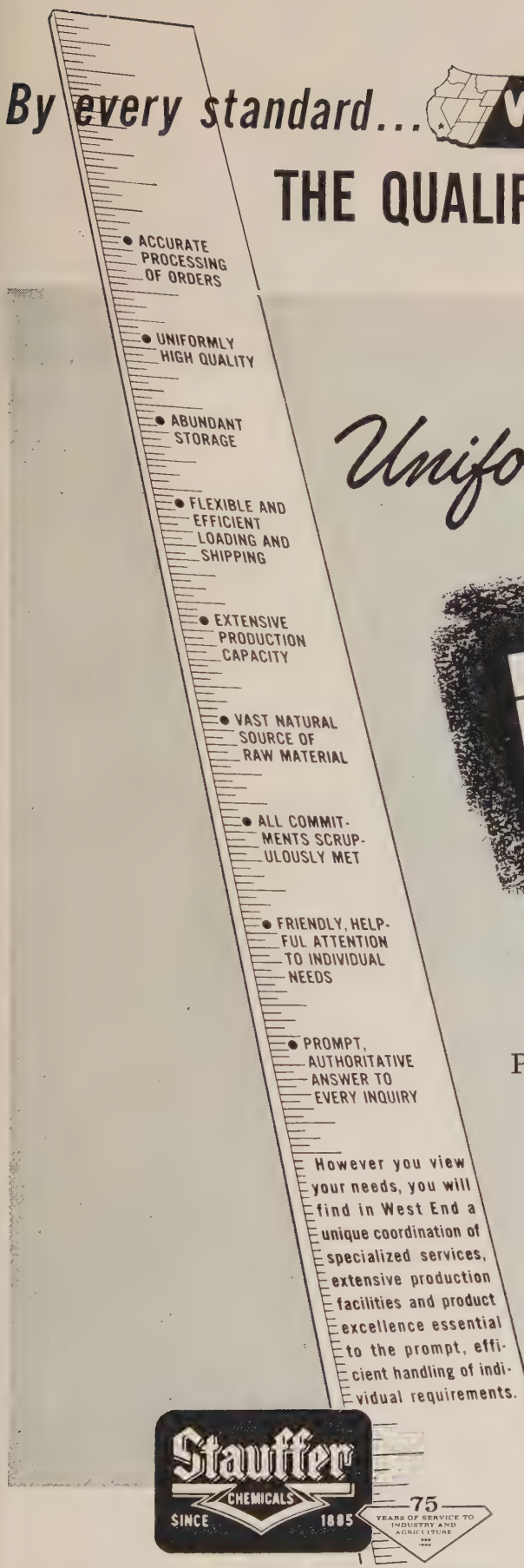
Table VII. Active Ingredients and Suppliers of all Materials Tested

Biostatic agent	*Active ingredients*	*Supplier*
A-36	Phenylmercuric acetate and colloidal silver	U. S. Movidyn Corp.
ATM-50	*N*-Alkylbenzyl-*N*,*N*,*N*-trimethylammonium chloride	Oronite Chemical Co.
ATM-X3	Unknown	Oronite Chemical Co.
5-Acetoxymercurivanillin	...	The Institute of Paper Chemistry
Actamer	2,2′-Thiobis-(4,6-dichlorophenol)	Monsanto Chemical Co.
Advacide PMN	Phenylmercuric naphthenate	Advance Solvents and Chemical Corp.
Advacide PMO	Phenylmercuric oleate	Advance Solvents and Chemical Corp.
Aminoethyl ethanolamine	...	Olin Mathieson Chemical Corp.
Armac CD 50	Acetate salt of a coco-derived amine	Armour and Co.
Arquad 2C	Dilauryldimethylammonium chloride	Armour and Co.
Arquad 2C Oleate	Dilauryldimethylammonium chloride	Armour and Co.
Arquad 12	Dodecyltrimethylammonium chloride	Armour and Co.
Arquad 16	Palmityl (cetyl) trimethylammonium chloride	Armour and Co.
Arquad 18	Octadecyltrimethylammonium chloride	Armour and Co.
Arquad S	Trimethylammonium chloride	Armour and Co.
Arquad S-2C	Soya trimethyl and dicoco dimethylammonium chloride	Armour and Co.
BCD 955	Mixture of sodium salt of 3(1-hydroxyethylidene) 6-methyl 2-H pyran 2,4(3H) dione, sodium borate, and copper-8-benzazine	Milport Chemical Co.
Becco peracetic acid 40%	...	Food Machinery and Chemical Corp.
Benochlor 3C	Similar to Chloroben	The Chloroben Corp.
Benzyl Parasept	Benzyl ester of *p*-hydroxybenzoic acid	Heyden Chemical Co.
Bio 2273	*N*-Cyclohexyl dichloromaleimide	Food Machinery and Chemical Corp.
Bio 2274	*N*-Benzyldichloromaleimide	Food Machinery and Chemical Corp.
Bio 4174	*N*-Phenyldichloromaleimide	Food Machinery and Chemical Corp.
Bionol A	Mixture of alkyl-9-methylbenzylammonium chlorides	General Dyestuff Corp.
Bis-(2-chloroethyl)-formal	...	Olin Mathieson Chemical Corp.
3-Bromothianaphthene-1-dioxide	...	Food Machinery and Chemical Corp.
BSM-11	Phenylmercuric acetate and 2,4,6-trichlorophenate	Buckman Laboratories, Inc.
BSM-11B	Phenylmercuric acetate	Buckman Laboratories, Inc.
Buffen 30	Phenylmercuric acetate	Buckman Laboratories, Inc.
Busan	Trimethyloctadecenyl, trimethyloctadecadienyl ammonium chlorides, and 8-hydroxyquinoline	Buckman Laboratories, Inc.
Butrol	Phenylmercuric acetate and potassium *o*-phenylphenate	Buckman Laboratories, Inc.
Butrol 8	Phenylmercuric acetate and 8-hydroxyquinoline	Buckman Laboratories, Inc.
Butyl Parasept	Butyl ester of *p*-hydroxybenzoic acid	Heyden Chemical Co.
Cadminate	Cadmium succinate	Mallinckrodt Chemical Works
Calcium formate	...	Trojan Powder Co.
Caso-cide (Permacide AM10)	Lactoxymercuriphenylammonium lactate	Castle Overton, Inc.
Cetylpyridinium bromide	...	Gallowhur Chemical Corp.
Chemocide MSS	Sodium salt of hydroxymercurymethoxypropylcarbamylphenoxy acetic acid	Chemo Puro Mfg. Corp.
Chloroben	Mixture of trichlorobenzene and *o*-dichlorobenzene	The Chloroben Corp.
Chloro-isophorone	...	Olin Mathieson Chemical Corp.
2-Chloromercuri-thiophene	...	Jefferson Chemical Co., Inc.
4-Chlororesorcin	...	Pennsylvania Coal Products Co.
2-Chlorothianaphthene-1-dioxide	...	Food Machinery and Chemical Corp.
3-Chlorothianaphthene-1-dioxide	...	Food Machinery and Chemical Corp.
Copper pentachlorophenate	...	Monsanto Chemical Co.
Copper 3-phenylsalicylate	...	Dow Chemical Co.
Cunilate 2493	Solubilized copper 8-quinolinolate	Scientific Oil Compounding Co., Inc.
Cunimene D 2536	Dehydroabietyl ammonium phenoxide (asphalt base)	Scientific Oil Compounding Co., Inc.
Cunimene D 2601	Rosin amine pentachlorophenate	Scientific Oil Compounding Co., Inc.
Cunimene D 2607	Dehydroabietyl ammonium phenoxide	Scientific Oil Compounding Co., Inc.
DAC 559	Tetrachloroglycoluril	Diamond Alkali Co.
DHAS	Sodium salt of dehydroacetic acid	Dow Chemical Co.
2,3-Dibromothianaphthene-1-dioxide	...	Food Machinery and Chemical Corp.
2,3-Dichloro-2,3-dihydrothianaphthene-1-dioxide	...	Food Machinery and Chemical Corp.
Diphenylguanidium trichloroacetate	...	Olin Mathieson Chemical Corp.
Dowicide A	Sodium salt of *o*-phenylphenol	Dow Chemical Co.
Dowicide B	Sodium salt of 2,4,5-trichlorophenol	Dow Chemical Co.
Dowicide C	Sodium salt of 2-chloro-2-phenylphenol	Dow Chemical Co.
Dowicide D	Sodium salt of 2-chloro-4-phenylphenol	Dow Chemical Co.
Dowicide E	Sodium salt of 2-bromo-4-phenylphenol	Dow Chemical Co.
Dowicide F	Sodium salt of 2,3,4,6-tetrachlorophenol	Dow Chemical Co.
Dowicide G	Sodium salt of pentachlorophenol	Dow Chemical Co.
Dowicide H	Unknown	Dow Chemical Co.
Dowicide K5	Sodium salts of various methylene bis-chlorophenols	Dow Chemical Co.
Dowicide S	Sodium salts of 2-chloro-6-phenylphenol and 2,3,4,6-tetrachlorophenol	Dow Chemical Co.
Dowicide 2S	2,4,6-Trichlorophenol	Dow Chemical Co.
Dowicide 31	Chloro-*o*-phenylphenol	Dow Chemical Co.
Drew liquid slime control	Phenylmercuric compound dissolved in glycolic acid and other solvents	E. F. Drew and Co.
Drew powder	Unknown	E. F. Drew and Co.
Duomeen C	Coco-derived diamine	Armour and Co.
Duomeen CDA 50	Coco-derived diamine and adipic acid	Armour and Co.
Dynacide	Phenylmercuric acetate and phenylmercuric chloride	William M. Stieh & Co.
Emulsept E607 special	*N*(acyl colaminoformylmethyl) pyridinium chloride	Merchants Chemical Co.
Ethyl formate	...	Olin Mathieson Chemical Corp.
Ethylmercuric methacrylate	...	Metalsalts Corp.
Ethyl Parasept	Ethyl ester of *p*-hydroxybenzoic acid	Heyden Chemical Co.
Experimental slimicide no. 5	3,5-Alkyl-tetrahydro-1,3,5-thiadiazine-2-thione	W. A. Cleary Corp.

By every standard... WEST END MEASURES UP AS THE QUALIFIED SUPPLIER OF SALT CAKE


• ACCURATE PROCESSING OF ORDERS
• UNIFORMLY HIGH QUALITY
• ABUNDANT STORAGE
• FLEXIBLE AND EFFICIENT LOADING AND SHIPPING
• EXTENSIVE PRODUCTION CAPACITY
• VAST NATURAL SOURCE OF RAW MATERIAL
• ALL COMMITMENTS SCRUPULOUSLY MET
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• PROMPT, AUTHORITATIVE ANSWER TO EVERY INQUIRY
However you view your needs, you will find in West End a unique coordination of specialized services, extensive production facilities and product excellence essential to the prompt, efficient handling of individual requirements.

Uniformly high quality

SALT CAKE

TYPICAL ANALYSIS

	Typical %	Guaranteed %
Na_2SO_4	99.57 -99.87	99.5 Min.
NaCl	.06 -.14	.2 Max.
B_2O_3	.03 -.06	.1 Max.
CO_2	.02 -.04	.05 Max.
Na_2O	.05 -.09	.1 Max.
H_2O	.01 -.10	.2 Max.
Insoluble	.001-.01	.05 Max.

Density: Struck Approx. 75 lbs./cu. ft.
Tapped Approx. 88 lbs./cu. ft.

Solution . . . Clear Color . . . White

Production techniques and controls developed at West End maintain the traditional quality of our product, even as we produce it in ever-increasing quantities. We submit West End as a chemical of highest purity and offer the above analysis for your consideration.

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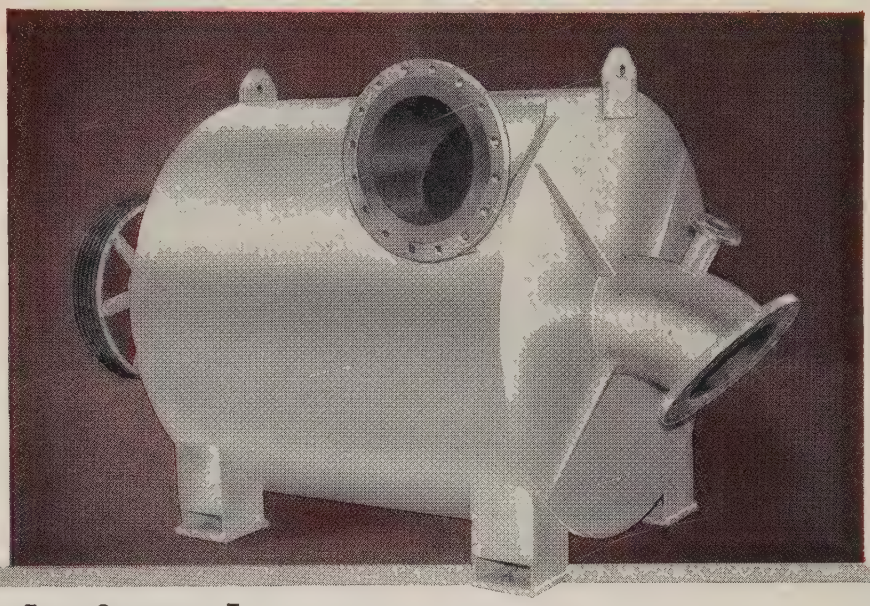
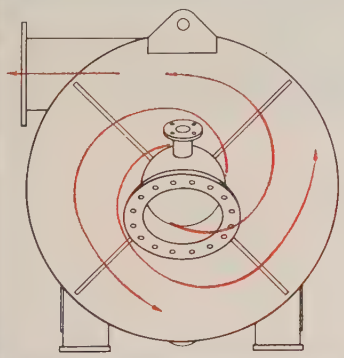
The DRAPER Felt
DRAPER BROTHERS COMPANY
CANTON, MASS.

Table VII. (*Continued*)

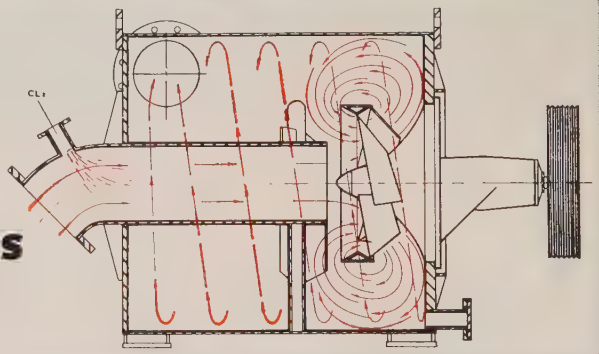
Biostatic agent	*Active ingredient*	*Supplier*
Formaldehyde-*p*-nitrophenyl hydrazone	. . .	Olin Mathieson Chemical Corp.
Fungicide 974 (CRAG)	3,5-Dimethyltetrahydro-1,3,5,2-H-thiadiazine-2-thione	Carbon and Carbide Chemicals Corp.
Fungitrol 50	Zinc naphthenate and dehydrobietylamine	Nuodex Products Co.
G4	2,2′-Dihydroxy-5,5′-dichlorodiphenylmethane	Givaudan Delawanna Inc.
Germycin-5	Phenylmercuric acetate	R. K. Laboratories Inc.
Gallicide	Phenylmercuric monoethanolammonium acetate	General Dyestuff Corp.
Gallosan	3,5-Dimethyltetrahydro-1,3,5,2-H-thiadiazine-2-thione	Gallowhur Chemical Corp.
Hydrazine difuroate	. . .	Olin Mathieson Chemical Corp.
5-Hydroxymercurivanillin	. . .	The Institute of Paper Chemistry
8-Hydroxyquinoline	. . .	Buckman Laboratories Inc.
Lignasan	Ethylmercuric phosphate	E. I. du Pont de Nemours & Co.
Merfenel	Phenylmercuric acetate	R. T. Vanderbilt Co., Inc.
Merfenel A (20% solution)	Phenylmercuric acetate	R. T. Vanderbilt Co., Inc.
Merfenel 51	Phenylmercuric acetate, sodium salt of dimethyl dithiocarbamic acid, and sodium salt of 2-mercapto-benzothiazole	R. T. Vanderbilt Co., Inc.
Mersynate	Phenylmercuric acetate and 2,4,6-trichlorophenol	Gallowhur Chemical Corp.
MetaSalt P	Diphenylmercuric ammonium propionate	Metalsalts Corp.
Meta-san 17	Phenylmercuric hydroxide	Metalsalts Corp.
Meta-san 35	Phenylmercuric chloride	Metalsalts Corp.
Meta-san E 100	Ethylmercuric oxyquinolinolate	Metalsalts Corp.
Meta-san Krylate	Phenylmercuric methacrylate	Metalsalts Corp.
Meta-san M 100	Methylmercuric oxyquinolinolate	Metalsalts Corp.
Meta-san P	Phenylmercuric propionate	Metalsalts Corp.
Meta-san PN	Phenylmercuric acetate and sodium nitrophenate	Metalsalts Corp.
Meta-san POS	8-Phenylmercuric hydroxyquinoline-5-sodium sulfonate	Metalsalts Corp.
Meta-san R	Phenylmercuric propionate	Metalsalts Corp.
Meta-san W	Phenylmercuric propionate	Metalsalts Corp.
Metasol A	Phenylmercuric acetate	Metalsalts Corp.
Metasol Krylate G	Phenylmercuric methacrylate	Metalsalts Corp.
Methylmercuric (2,4-dichlorophenoxy) acetate	. . .	Ringwood Chemical Corp.
Methylmercuric hydroxide	. . .	Ringwood Chemical Corp.
Methyl Parasept	Methyl ester of *p*-hydroxybenzoic acid	Heyden Chemical Co.
Microdyn	Colloidal silver and colloidal copper	U. S. Movidyn Corp.
Monobutyl *m*-cresol	. . .	Koppers Company, Inc.
Movidyn	Colloidal silver	U. S. Movidyn Corp.
NC 1235	1,3-Di-nonylbenzimidazolium bromide	Nepera Chemical Co., Inc.
NC 2050	2-Thiocyanopyridine oxide	Nepera Chemical Co., Inc.
Negant 30 s	Phenylmercuric acetate	Germicidal Corp. of America
NP 1711	Tetrachloromethylene cyclopentenedione-1,3	Pennsylvania Salt Mfg. Co.
NP 1713	Octachloromethylene-1-cyclopentene-2	Pennsylvania Salt Mfg. Co.
Nacconol FSNO	. . .	National Aniline Division, Allied Chemical & Dye Corp.
Nalco 21	Unknown	National Aluminate Co.
Nalco 21B	Mixture of saturated and unsaturated chlorophenols	National Aluminate Co.
Nalco 21M	Sodium salt of pentachlorophenol	National Aluminate Co.
Nalco 21S	Mixture of sodium 2,4,5-trichlorophenate and sodium pentachlorophenate	National Aluminate Co.
Nalco 23	Unknown	National Aluminate Co.
Nalco 234	1-(2-hydroxy ethyl)-1-benzyl-2-tridecyl imidazolinium	National Aluminate Co.
No Slyme	3,5-Dimethyl-tetrahydro-1,3,5,2-H-thiadiazine-2-thione	W. A. Cleary Corp.
Nuodex copper HOH-6%	Copper ammonium naphthenate	Nuodex Products Co., Inc.
Nuosept	Ethyl ester of *p*-hydroxybenzoic acid, *n*-propyl ester of *p*-hydroxybenzoic acid, and 2,2′-methylene bis-(4-chlorophenol)	Nuodex Products Co., Inc.
Nuozene	2,4-Dichloro-6-(*o*-chloroanilino)-*s*-triazine	Nuodex Products Co., Inc.
Nytron	Sulfonated ketones, amines, alkyl sulfamates, alkylidene sulfamates, and alkylidene sulfamate bisulphite	Solvay Process Co.
Ottacide	Blend of *p*-nitrophenol, *p*-chloro-*meta*-xylenol, and tetrachlorophenol	Ottawa Chemical Co.
Ottafect	2-*tert*-Butyl-4-chloro-5-methylphenol	Ottawa Chemical Co.
Ottasept 430	*p*-Chloro-*meta*-xylenol	Ottawa Chemical Co.
P1006	Nonmetallic ketone	Gallowhur Chemical Co.

Sandy Hill KAMYR LOW DENSITY MIXER





designed for continuous blending at consistencies of 1 to 5%



USES:

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- For blending two or more stocks
- For mixing chemicals or other additives

The Sandy Hill-Kamyr Low Density Mixer has a proved record for superior blending.

The short mixing cycle results in rapid, more thorough mixing.

This low density mixer combines a Kamyr CR-4 circulation pump in a specially designed tank type vessel. Materials of construction are specified to meet the operating conditions. Write or phone for complete details.

THE SANDY HILL IRON AND BRASS WORKS
HUDSON FALLS, N. Y.

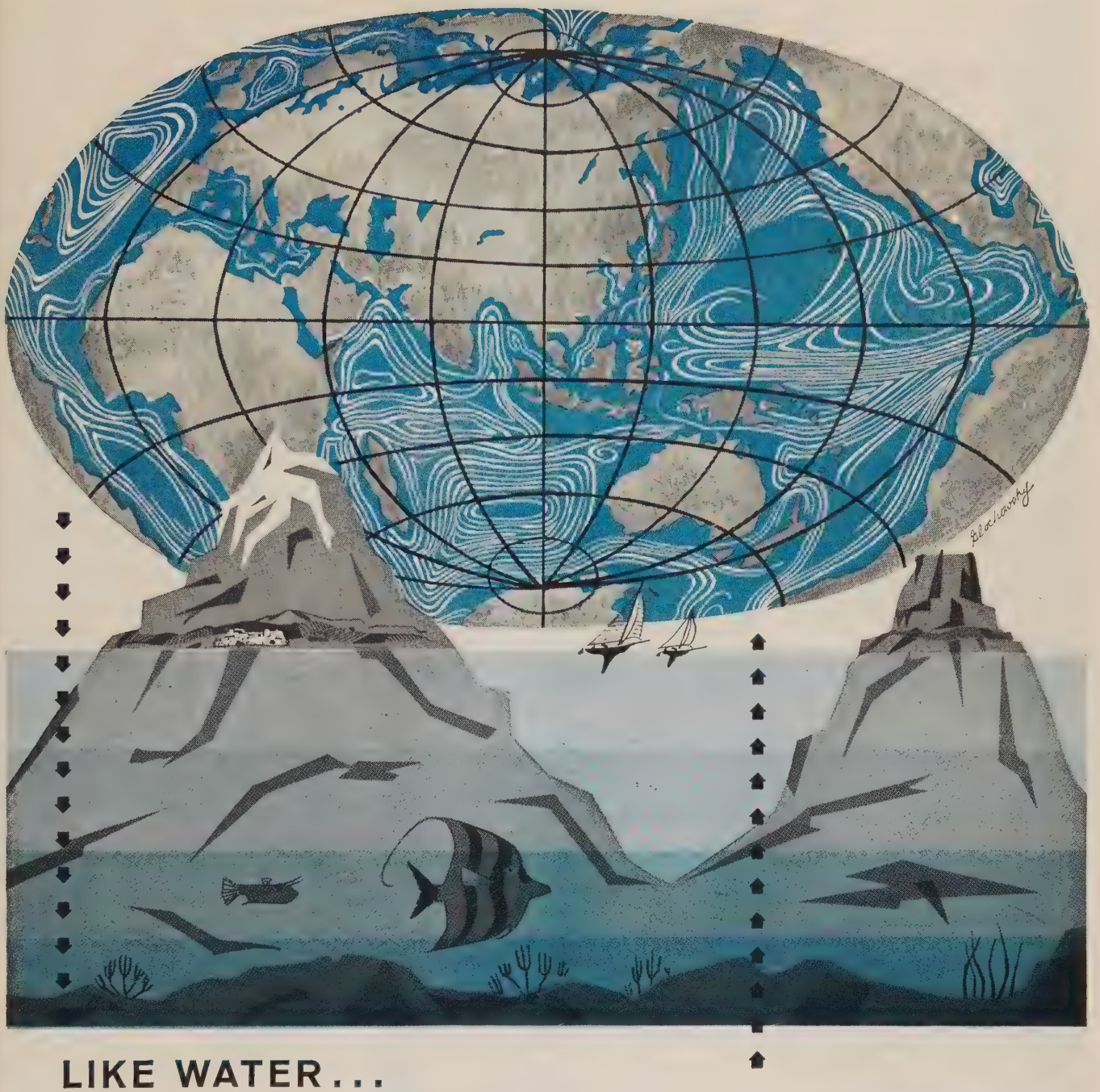
Table VII. *Continued)*

Biostatic agent	*Active ingredients*	*Supplier*
P200A	Phenylmercuric acetate	Gallowhur Chemical Co.
P.M. 32 Powder	Phenylmercuric acetate	Falls Chemical Products Co.
PMA	Phenylmercuric acetate	W. A. Cleary Corp.
PMAS	Phenylmercuric acetate	W. A. Cleary Corp.
Penetox	Unknown	Magnus Chemical Co.
Penn Salt C. A. F.	Copper ammonium fluoride	Pennsylvania Salt Mfg. Co.
Perma Cide 135	Phenylmercuric acetate	Refined Products Corp.
Perma Cide AL-10	Phenylmercuric lactate	Refined Products Corp.
Permatox	Unknown	Chapman & Co., Inc.
Permycin-5	Hydroxy mercury amino salt	R. K. Laboratories, Inc.
Permycin C-5	Chloro mercury amino salt	R. K. Laboratories, Inc.
Phenylmercuric undecylinate	. . .	Metalsalts Corp.
Phymer	Unknown	Wood Treating Chemicals Co.
Polymyxin B sulfate	. . .	Charles Pfizer and Co., Inc.
Preventol GD	2,2′-Dihydroxy-5,5′-dichloro-diphenylmethane	General Dyestuff Corp.
Preventol GDC	2,2′-Dihydroxy-5,5′-dichloro-diphenylmethane	General Dyestuff Corp.
Propargyl-*N*-phenyl carbamate	. . .	Olin Mathieson Chemical Corp.
Propyl Parasept	Propyl ester of *p*-hydroxybenzoic acid	Heyden Chemical Co.
Purasan AD	Alkyldimethylbenzylammonium chlorides	Gallowhur Chemical Corp.
Purasan DC	Alkyldimethyl-3,4-dichlorobenzylammonium chlorides	Gallowhur Chemical Corp.
Purasan X-3	Unknown	Gallowhur Chemical Corp.
Puratized 300	Phenylaminocadmium dilactate	Gallowhur Chemical Corp.
Puratized 641	Phenylmercuric formamide	Gallowhur Chemical Corp.
Puratized 1122	Heterocyclic quaternary ammonium derivative	Gallowhur Chemical Corp.
Puratized F	*p*-Aminophenylmercuric acetate in lactic acid	Gallowhur Chemical Corp.
Puratized N5DS	Phenylmercuric triethanolammonium lactate	Gallowhur Chemical Corp.
Puratized N5E	Phenylmercuric triethanolammonium lactate	Gallowhur Chemical Corp.
Puratized SC	Phenylmercuric lactate	Gallowhur Chemical Corp.
Pyridylmercuric acetate	. . .	Mallinckrodt Chemical Works
Pyridylmercuric chloride	. . .	Mallinckrodt Chemical Works
Quicksan EM	Phenylmercuric acetate and ethylmercuric acetate	Stecker Chemicals, Inc.
Ramplex C 100	Unknown	Merchants Chemical Co.
Roccal	Mixture of alkyldimethylbenzylammonium chlorides	Winthrop Chemical Co., Inc.
RD 1652	Sodium salt of didodecyldithiocarbamic acid	Armour and Co.
Rosin-Amine D Acetate	. . .	Hercules Powder Co.
Santobrite	Sodium salt of pentachlorophenol	Monsanto Chemical Co.
Santophen 1	*o*-Benzyl-*p*-chlorophenol	Monsanto Chemical Co.
SC 100	Phenylmercuric acetate	Stecker Chemicals, Inc.
SC 200	Phenylmercuric acetate	Stecker Chemicals, Inc.
SCXP 1170	2-Bromothianaphthene-1-dioxide	Food Machinery and Chemical Corp.
SCXP 1290	2,3-Dibromo-2,3-dihydrothianaphthene-1-dioxide	Food Machinery and Chemical Corp.
S-ethyl mercury thioammeline	. . .	Olin Mathieson Chemical Corp.
Slime-Acide L.M.	Quaternary ammonium organomercurial complex	Johaneson Wales & Sparre, Inc.
Slime-Acide M	Phenylmercuric acetate	Johaneson Wales & Sparre, Inc.
Slime-trol RX3	Copper sulfate and sodium pentachlorophenate	W. H. and L. D. Betz Company
Slimicide MSC	Unknown	Wood Treating Chemicals Co.
Slimodyn	Phenylmercuric acetate, colloidal silver, and colloidal copper	U. S. Movidyn Corp.
Sodemmet	8-Ethylmercuric hydroxyquinoline-5-sodium sulfonate	Metalsalts Corp.
Sodium salt 2-pyridinethione	. . .	Olin Mathieson Chemical Corp.
2,4,6-Trichlororesorcinol	. . .	Pennsylvania Coal Products Co.
Tris-*beta*-chloroethyl phosphate	. . .	Olin Mathieson Chemical Corp.
Tween 80	Nonionic fatty acid ester of sorbitol	Atlas Powder Co.
Tyrothricin	. . .	S. B. Penick & Co.
USF 188	Sodium alkyl phenates	United States Fungicide Co.
Vancide 32	Beta-hydroxyethylpyridinium salt of 2-mercapto-benzothiazole	R. T. Vanderbilt Co., Inc.
Vancide 51	Sodium salt dimethyl dithiocarbamic acid and sodium salt of 2-mercaptobenzothiazole	R. T. Vanderbilt Co., Inc.
Vancide F-999	Octachlorocyclohexane	R. T. Vanderbilt Co., Inc.
Zonemask	Quaternary ammonium compound	Murphy Chemical Co.
Zonemask L-39	Quaternary ammonium compound	Murphy Chemical Co.

pliers of the materials are listed in Table VII. The inhibiting concentrations are based on a w/w basis of the compounds as received. Although it is a requirement that the nature of the active ingredients be disclosed before a compound will be included in this program, it is not required that the exact formulations of the material be revealed. Therefore, in many instances, the percentage of active ingredient in a compound is unknown and a conversion of the data to an active ingredient basis is not possible.

There has been no attempt made to rate or rank the materials on the basis of their performance toward the individual or the combined test organisms. It should be kept in mind that the laboratory procedures used in obtaining these data are limited by the fact that the test conditions differ greatly from those encountered in industrial usage. The data do not include the necessary considerations end usage, toxicology, solubility, cost, etc.; therefore, such a ranking would have little more value that the present data form. It is equally true, however, that to attempt to test such a large number of compounds on a use-condition basis would be an enormous, if not impossible, task. These data serve as an index to the biological activity of the material and permit a more intelligent selection of possible candidate test materials for a particular use than an arbitrary selection. It is suggested that for a more critical evaluation of a compound's merits the material should be tested under conditions which more closely approximate those of actual use.

For those interested in submitting materials for testing under this program the committee will accept for evaluation those materials offered to the pulp and paper industry for slime control and the preservation of



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coatings of Ethylex® Gums seek their own level

Perfectly smooth sheets are assured when you use Ethylex Gums in your coatings. How? The superior water-retention properties of Ethylex Gums allow time for coatings to level, minimizing surface irregularities and imperfections. The result? A smoother, more uniform surface with high pick values, excellent printability and good varnish holdout.

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FOR ALL PAPER COATINGS

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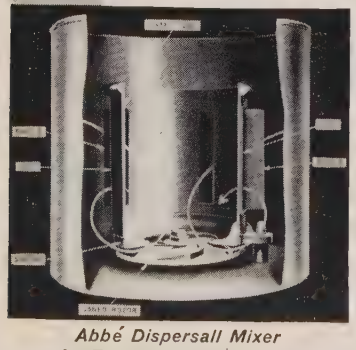

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pulps, sizing materials, felts, finished papers, etc. Experimental materials which have properties indicating a potential use in microbiological control in our industry are also acceptable; however, the nature of the active ingredient(s) of all materials submitted for testing must be revealed.

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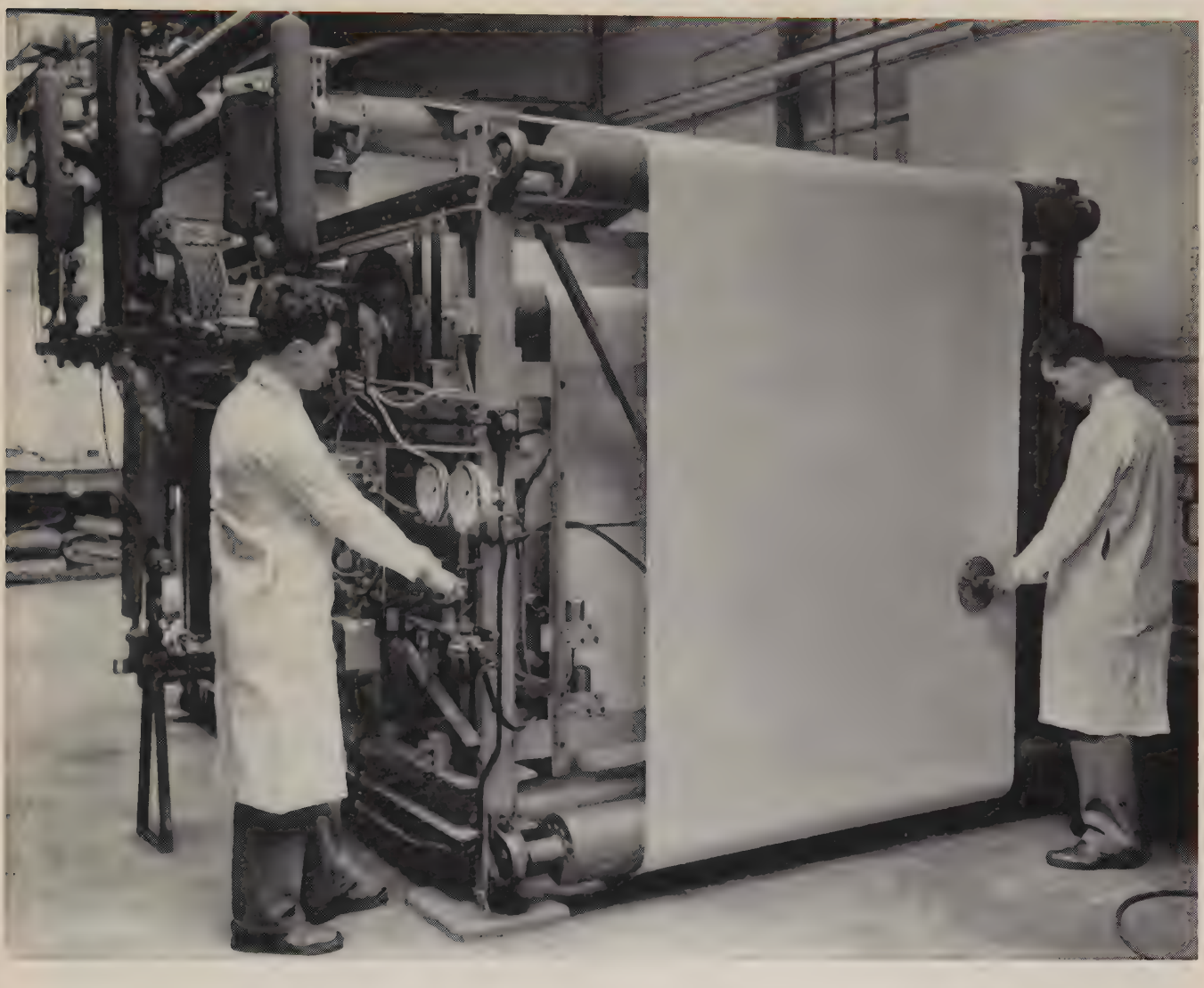

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European Approach to Printability Research

MARVIN C. ROGERS

Two much overworked words in the language of graphic arts spokesmen are "research" and "new developments." There is almost complete agreement that research and development are keys to the future, but to the printing industry, they apply more specifically to the belief that it is the supplier (inkmakers, papermakers, machine builders) to whom research is most important, and it is he on whom the responsibility for research rests. A common attitude may be expressed by the comment, "it is the supplier who has most to gain from research. He will get his return in the price of the product he sells."

In discussions with the same industry spokesmen and with industry leaders, we have found another area of agreement between them. They have been unanimous in their insistence that government should have no part in a program of industry research and development, at least where graphic arts is concerned. This attitude toward government is understandable in view of the widespread publicity given to government projects during the past 25 years. The fact that government is influenced by business and individual interests of citizens is never admitted. Neither is the fact that scientists and other professionally trained government workers are devoted to their job assignments to a degree not unlike the devotion of the teacher and professor. One seldom hears of scandals and waste among the technical and professional government employees.

Of course, there is no immediate pressure for profits present in government research, but the lack of profit motive has little to do with the need for basic information from fundamental studies so badly needed in the graphic arts or the zeal with which workers attack the problems. There is little doubt that any qualified research team attacking the fundamental problems of the graphic arts would get useable results in reasonable periods of time. It would have to be given the opportunity, however.

Business and other leaders too frequently generalize regarding the practices of government, when they as citizens are responsible for its activities. It is not difficult to find instances in industry activities where the "government work" principle is practiced.

These comments may seem foreign to this discussion, but they form an excellent background for the discussion of European approach to printability (printing) research. Some of the most fascinating research work under way in the graphic arts is that going on in the European institutes where industry and government in most instances have teamed together to look for the answers to industries' problems. We in the United States are unique in our insistence that research work must be supported only by "free enterprise," and yet our government is probably the world's biggest spender for research. Its spending is related almost entirely to national defense, agriculture, mining, and health, and little attention goes to other industries.

There is no organized plan for wide-coverage industrial research in the United States, where government would participate in or encourage it, except those instances mentioned above. It is doubtful if many industries have given this much thought, because of the competitive problems involved. It is doubtful too, if any industry could do better than the paper industry with its Institute of Paper Chemistry, even if there were government support. A large portion of industrial research deals with defensive programs designed to get out of trouble with customers; much of what is left is gadgetering and design changes. In the graphic arts industry, the Lithographic Technical Foundation is an excellent example of a segment with specific problems, getting together on a program designed to be practical. There are others such as the inkmakers, photoengravers, electrotypers, and gravure printers who have formed specialized programs to aid their peculiar interests. With few exceptions, the inkmakers being one, these programs in the graphic arts are designed to yield practical operating results, and only a minimum of fundamental research work is carried on.

Much of the printability research work at Lehigh University is fundamental in character, and it illustrates the type of study needed by industry for its future progress. This work cannot be aimed at the goal of dollars tomorrow; but only at the goal of producing fundamental information to the industry on which the design of future materials and machines may be based.

One of this writer's most satisfying experiences in graphic arts work has been participation in the International Conferences of Printing Research Institutes, usually held in Europe. Research workers from a dozen or more countries meet in these conferences to discuss the studies and experiments in which they are engaged. The majority of the participants represent laboratories in Europe where research is partially sponsored; or encouraged in some way by government agencies. Usually the principal support comes from subscriptions from industry members, and their programs are related to improved processes, machines, or materials. Buildings in a few cases were built by government, and leased to the associations.

It is on these groups and their activities that we place emphasis in this paper, because it appears to this writer that the European approach to printing research may be characterized by cooperation, philosophy, and attitudes. One aspect of the philosophy is exemplified in the attitudes of several governments toward the necessity to encourage research in many fields of industrial activity. A second aspect is the stress placed on fundamental work by the industry repre-

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Gravure Coating-Direct	Lacquer, Adhesives, Coloring
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Kiss Coating with Metering Bar	Latex, Carbons, Clay
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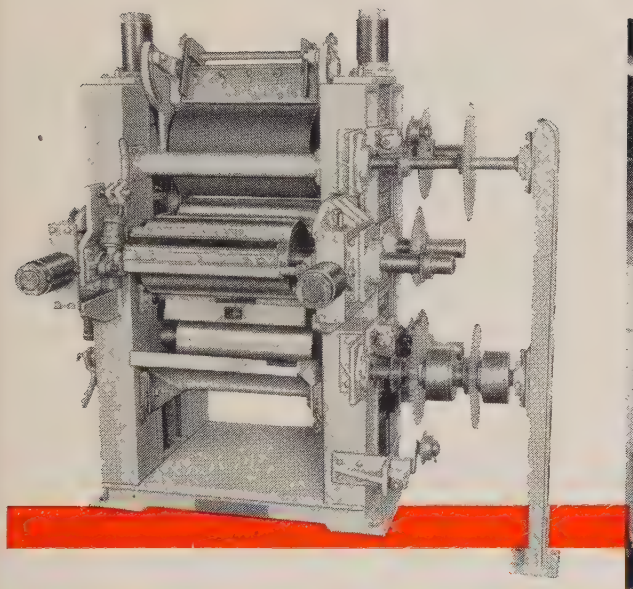

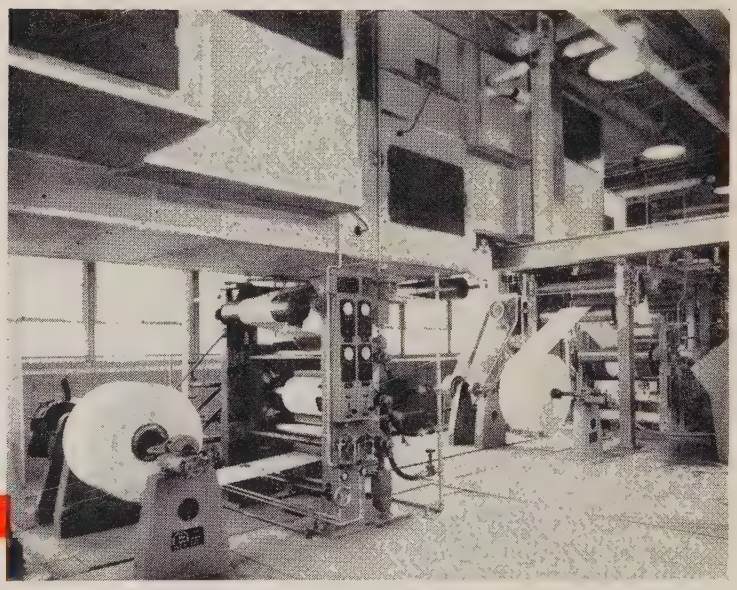

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sentatives and the scientific workers, themselves. Scott (W. S. Cowell, Ltd.) in speaking before the International Federation of Master Printers at their 1957 Meeting in Lausanne had this to say: "...research associations have three main activities: the first is trouble shooting. ...but it must be kept within limits. The second is research on recognized problems —applied research...it must have value to industry. ...the third is fundamental or basic research, and has no immediate application. ...One cannot match each piece of basic research neatly with its application, for a great part of scientific knowledge had no industrial application so far perceived."

Each research organization has its special strengths, either in the nature of the organization or of the individuals within its laboratories. An excellent example of industry approach to printing research is that in Great Britain; the Printing, Packaging, and Allied Trades Research Association—known as PATRA, located at Leatherhead, just outside London. The organization has modern and well-equipped laboratories, employing nearly 100 people. There are 700 printing and supply and over 200 packaging firms subscribing according to the size of the firm. In addition, there is the annual grant from the Department of Scientific and Industrial Research.

PATRA's Research Committee consists of printers, packaging experts, materials and machinery suppliers, and this group has the job of initiating a research program and reviewing its progress. Among the efforts of a fundamental nature being studied are the phenomena taking place when inked type meets paper, a printability study not unlike those we find in the laboratories of some United States ink and paper suppliers. The problem is fundamental and of basic interest to every printer, papermaker, and ink or press manufacturer.

It is likely that the impetus given to research and development by the government grant has done much to build up the strength of PATRA, but it was the enthusiasm of the industry leaders themselves which gave the organization a start just 30 years ago.

Great Britain is not alone in graphic arts research. Well established and well equipped laboratories can be found in Sweden and the Netherlands, Germany, and elsewhere, where much of the research work is coordinated through state research agencies. These agencies provide the broad technical background facilities necessary to any research program, without the necessity for any single industry to support extra personnel. This situation is not unlike the larger research institutes and foundations operating in many industrial areas here. West and East Germany each has functioning, well-staffed laboratories; and Norway and Finland in which there are institutes devoted primarily to the study of pulp and paper, the printing uses for paper are considered in projects related to the printability of paper.

There are printing research laboratories in Switzerland, France, Italy, Austria, and the U.S.S.R. Most of the programs are small by United States standards, but when one considers the physical size and populations of these smaller countries, it is gratifying to find them supporting fundamental activity not generally tolerated by the printing industry here.

The Swedish Graphic Arts Laboratory is located in a modern building in Stockholm, adjacent to the Pulp and Paper Research Laboratories. Much basic work has been done there on the printability of paper. The work is justified by the dependence of the Swedish economy on pulp, but the laboratory is not confined to work of a narrow field. Many of the projects are fundamentally related to the problem of relating paper printability to paper characteristics.

Work in Norway and in Finland relates to the properties of pulps and paper. These countries are not major printing centers, and their export economy depends on pulp from wood. Both have well-equipped laboratories and their staffs have made printing processes study an important part of their work.

In Denmark, research is well organized, but the approach is different. Newsprint users conduct their own study which is designed to raise the quality of the newsprint used by them. They are also interested in test methods designed to maintain quality. Perhaps this is due to the import nature of the industry. It is in the Graphic College of Denmark, however, where training and research are partially combined. The staff is small, but the fact that there is training and research in a special institute, in a country as small as Denmark, is a tribute to the Danish people.

The German tradition for research is so well known that it needs little amplification here. The German scientist historically has been dissatisfied with processes as they are, and has constantly sought ways to bring about improvements. Printing firms of West Germany have a unique organization operating at Munich. The German Institute for Research in Graphic Arts

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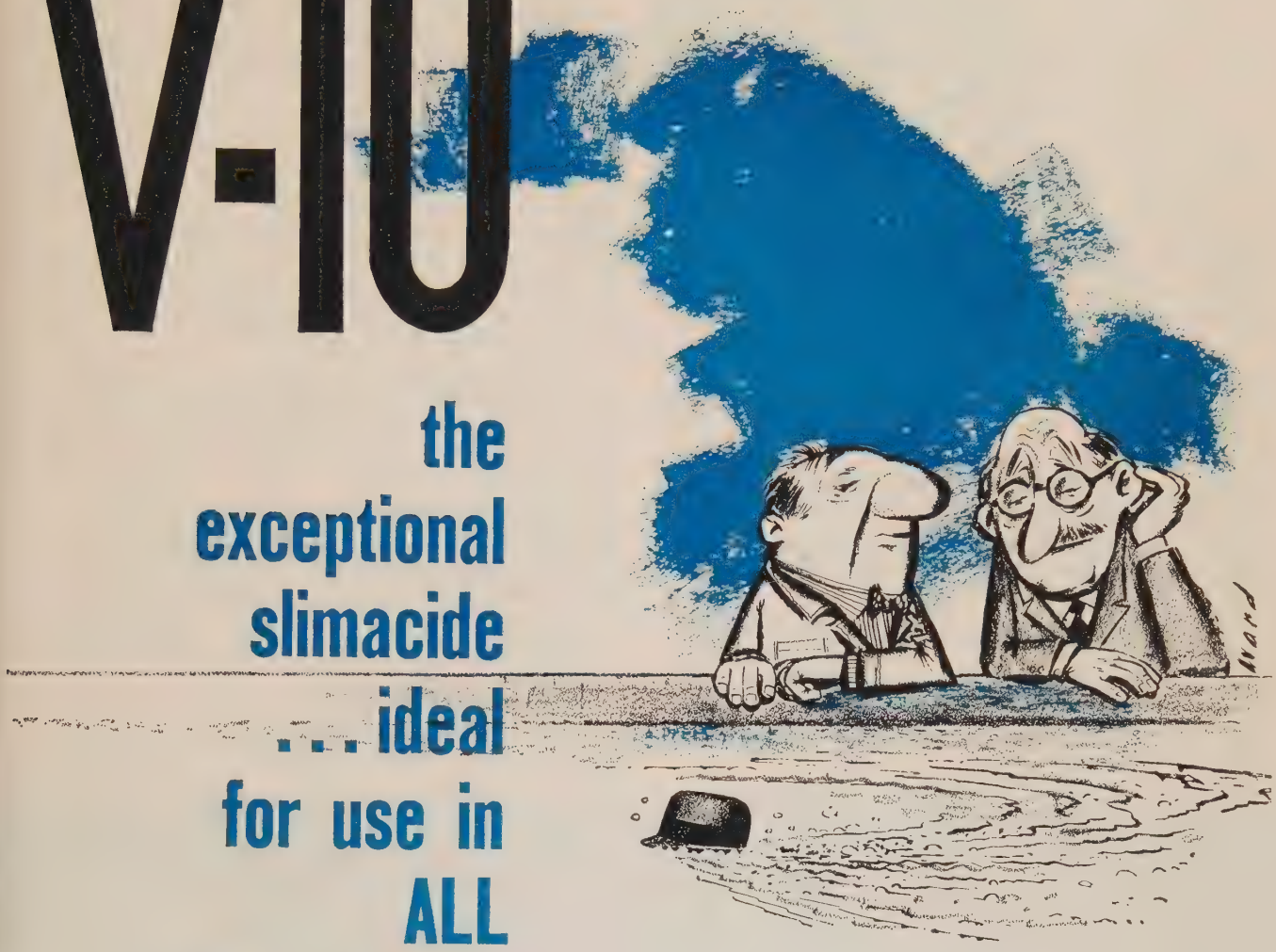

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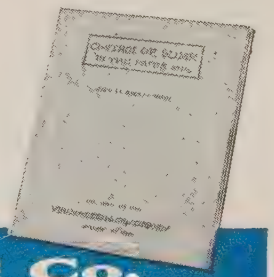

* *Test results available on request.*

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gives the visitor the feeling that he is in the midst of a graphic science, rather than graphic arts. Located in Bamberger Haus in Munich, it is a showplace as well as a scientific institute. The sponsoring group is made up of members of the printing industry in Germany, but the institute gets some support from foreign organizations as well.

In the program of study, have been comprehensive researches on printability, color reproduction, instrumentation, and the plate-making processes.

Other German research activities are at Darmstadt where the equipment makers sponsor a research program, and at Leipzig, in East Germany. Both of these organizations operate under the direction of capable and trained scientists.

Programs in industry supported institutes of France, Italy, Switzerland, and Netherlands are each of a scientific nature although testing and specifications have importance where large imports are concerned. The Dutch scientists have been successful in developing a printability tester (IGT tester) which is rapidly finding application in the United States. The work of Tollenaar on Moire' pattern in multicolor printing, has recently become a publication of the Research and Engineering Council of the Graphic Arts.

Although publications and information on the activities in the institutes behind the Iron Curtain are not plentiful, there is good indication that much organized activity is being carried on in U.S.S.R. and elsewhere. It does not appear to have been as extensive as in other countries.

From this review it should be clear that even though governments participate to a degree in the work of the European printing research institutes, their functions are primarily those of encouragement and grants-in-aid to industries which help themselves. Fundamental study has been given the lift it needs.

We have implied at least, that fundamental studies are more in evidence throughout Europe than in the United States. This is true, but only because there are more different organizations available to carry on this type of work. We said too, that attitudes were important to the success of the European programs. These attitudes are those of the staff members in the institutes, and of the industry members with whom they come in contact.

At the second conference of printing research institutes, Poulter of Great Britain surveyed the printability literature and found that of the 70 references to printability, more than 50 came from United States or Canadian publications. They tended to group themselves into two methods of approval: (1) proof press methods—visual judging of prints, and (2) physical tests, correlated with printing results.

These methods of approach are still much in evidence in printability work here or in Europe, and they serve specific functions quite well. However, those methods yielding the best results appear to be those requiring the greatest amount of interpretation and the greatest amount of time. The proof press appeals to many workers because it resembles production methods.

Where physical tests have been studied, they have been those mill tests—opacity-smoothness—gloss—and compressibility—for which methods and equipment are readily available. Broad correlation has been impossible to attain, but there are isolated situations in which correlations have been satisfactory.

Precision press equipment was not available to the European worker, and he was forced to resort to his ingenuity to develop laboratory instruments from which he might obtain the data he desired. Hence, much work relating to printability covers only a small area, and is not directly applicable to printing problems. Absorption rates have received a considerable amount of attention and are part of the procedures still in favor by some workers. They are part of current active printability work in the United States. It is likely that this need for equipment, or the fact that they did not have it, has been responsible for the semibasic work being done. We cannot review all of it, but here are a few examples:

Tollenaar at the Dutch Institute for instance—has devoted attention to "Porosity as It Relates to Printability" and has tried to analyze the influence of capillary properties to the phenomenon of ink printing on paper.

Ohlsson and Pihl (Swedish Graphic Arts Laboratory) in their work "Relations between Paper and Ink" built a simulated "press" in which they could print from a plate having a known amount of ink and determine the degree of transfer from the plate to the paper. This technique has found wide application elsewhere.

These workers were also concerned with the relationship between oil penetration and porosity, and found as others have found that the correlation is not good. Workers in Denmark have devoted much attention to the correlations of test instrument results with printability.

Another approach to printability is that taken by Coupe (PATRA) in Great Britain when he tried to determine "Influence of Chemical Residues on Printability." He considers the influence of such things as reducible sulfur on printing of image or other pigments likely to be affected by this material.

In still other approaches, the Dutch in simulating printing, have tried to approach practical printing with an instrument susceptible to precise control. The IGT tester has resulted from this work, and a German instrument is also available, which functions in a similar manner.

If this review were to be complete, many other references would be included. Certainly, the work of Albrecht and Schirmer on "Printing Ink Printability," and that of Rupp on "Wetability of Paper by Water and Ink" would have to be mentioned.

In spite of the fundamental approach, there is the desire to be practical, and to apply the studies to practical testing of paper for printability. The main problem seems related to the widely varying requirements of the different industries and the fact that fundamental work is still too sparse to permit broad correlation. It would be extremely helpful to printability research generally, if there were a central research group financed by the printing industry to get at these problems in the United States. Perhaps more of the current efforts in that direction will meet with more success.

Received July 12, 1960. Presented at the Eleventh Coating Conference of the Technical Association of the Pulp and Paper Industry, held in Chicago, Ill., May 23–25, 1960.

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Relative Toxicity of Biostatic Agents Suggested for Use in the Pulp and Paper Industry 1959 Supplement

J. H. CONKEY and J. A. CARLSON

THE Biological Control Committee of the American Paper and Pulp Association sponsors at The Institute of Paper Chemistry, as a part of its general program of biological research, a laboratory investigation of the relative toxicity of various biostatic materials offered to the pulp and paper industry. This program was initiated in 1941 for the purpose of providing the industry with comparative toxicity data on commercially available slime-control agents. The program has since been extended to cover materials suggested for uses other than slime control and certain experimental materials believed to be of potential value in our area.

A policy has been established by the Biological Control Committee of publishing a complete review of the data obtained under this program at five-year intervals with current work presented as yearly supplements. This report presents the data of the screening tests completed during 1958 and is the first supplement to the review report published in 1958 (*1*).

The laboratory method used to determine the relative toxicity of a test material may be found in the 1958 review report (*1*). Briefly, it is an agar-Petri plate procedure using as test organisms *Aerobacter aerogenes*, *Bacillus mycoides*, *Aspergillus niger*, and *Penicillium expansum*. The 33 materials covered in this report were obtained directly from their suppliers or were sent to us by pulp or paper mill personnel desiring information regarding their toxicity. The inhibiting concentrations found toward the four test organisms are presented in Table I and the active ingredients and the suppliers of the materials are listed in Table II. (See page 42 A)

It should be kept in mind that the laboratory procedures used in obtaining these data are limited by the fact that the test conditions differ greatly from those encountered in industrial applications. The inhibiting concentrations serve only as an index to the biological activity of the material and, therefore, to obtain a more critical evaluation of a material it should be re-examined under conditions which more closely approximate those of actual use.

For those interested in submitting materials for testing under this program, the committee will accept for evaluation those materials offered to the pulp and paper industry for slime control and the preservation of pulps, sizing materials, felts, finished papers, etc. Experimental materials which have properties indicating potential usefulness toward microbiological control in the industry are also acceptable; however, the nature of the active ingredient(s) of all materials submitted must be revealed.

J. H. CONKEY, Research Aide, and J. A. CARLSON, Research Aide, The Institute of Paper Chemistry, Appleton, Wis.

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Table I. Relative Toxicity

Biostatic agent	*Inhibiting concentration, p.p.m.* Aerobacter aerogenes	Bacillus mycoides	Aspergillus niger	Penicillium expansum
Busan 881	950	30	1500	300
Chemocide CAA	>3000	3000	1500	550
Chemocide CETB	45	3	>3000	1500
Chemocide ETA	7	0.4	3	2
Chemocide HK	>3000	6	>3000	>3000
Chemocide LPL	>3000	200	>3000	>3000
Chemocide PCMC	200	200	150	75
Chemocide PCPD	1500	750	2000	1500
Chemocide SA	55	65	65	50
Chemocide XK	2500	15	>3000	150
Dimethylamine borane	>3000	1500	1500	1500
Dodecyl boric acid	>3000	15	>3000	>3000
Fungi-Ban	80	4	20	40
Merfenel 51[a]	150	0.3	20	3
Merfenel 41	30	2	300	70
Meta-san D-245	3	0.3	4	3
Meta-san D-246	2	0.3	4	3
Meta-san DM	3	0.1	5	2
Meta-san D-OH	5	0.9	20	10
Meta-sol L	15	0.9	40	25
Nalco 201	50	9	35	65
Nalco 236	450	150	>3000	>3000
Nonyl boric acid	350	85	2500	2500
Pyridine borane	350	150	550	400
Quicksan C-10	30	6	150	45
TBTO	30	1	15	25
Thienylmercuric acetate	20	1	150	20
Tin Anti-Slime	90	4	25	30
p-Toluenesulfonamide	>3000	>3000	>3000	2500
Trimethylamine borane	>3000	>3000	>3000	>3000
Vancide 26 EC	250	40	1500	250
Vancide 51 Z	60	0.9	>3000	2000
Vancide 89	150	20	>3000	85

[a] Reacted form—previous tests (*1*) made on blended product.

THIS SLOW-MOTION ATLANTIC TESTING MACHINE

CAN IMPROVE YOUR HIGH-SPEED PACKAGING OPERATIONS

At the rate that paraffin coated boxes and cartons slide from stack to packaging machine, even a split second of delay can start a costly, time-consuming chain reaction all along the line. To insure continuous and profitable operation, the waxed surfaces must slip freely across each other during their instant of contact.

Shown above is an exclusive testing device used by Atlantic research technicians to determine slip properties of paraffin waxes. Basically, the test consists of measuring the force necessary to slide one paraffin coated sample along another. Slip is reported in terms of the coefficient of friction, with low coefficients as the objective. The slip characteristics of a proposed paraffin coating can thus be determined before production gets underway.

This Slip Test Machine is typical of the testing equipment used in the Atlantic Research Department. Others include our exclusive Imflex Tester for determining impact resistance and flexibility . . . the Seal Strength Tester which measures the force necessary to separate wax laminations and seals . . . the Beam Test, to establish durability of waxed corrugated stock . . . and tests for gloss, tensile strength and resistance to grease.

Your experienced Atlantic Sales Engineer will be happy to give you complete information on how our paraffin research facilities can be of service to you. In many cases he can supply on-the-spot assistance. When more specialized information is required, he has the full and prompt assistance of the Atlantic Research and Development Department.

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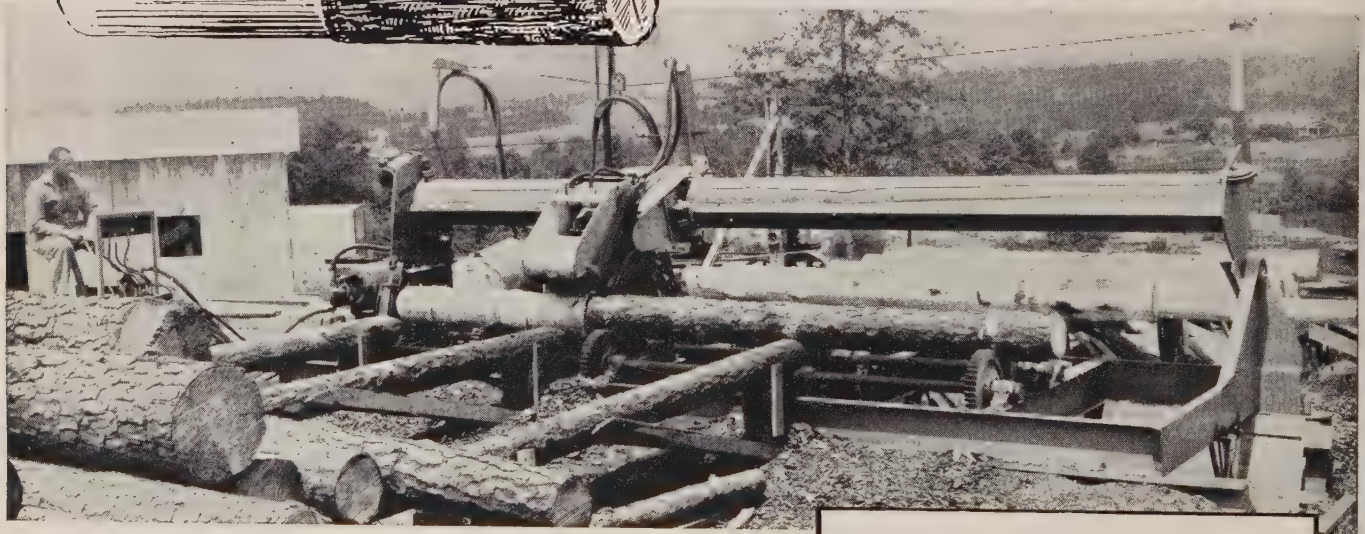
Table II. Active Ingredients and Suppliers of Test Materials

Biostatic agent	Active ingredients	Supplier
Busan 881	Disodium cyanodithioamidocarbonate and potassium *N*-methyldithiocarbonate	Buckman Laboratories
Chemocide CAA	Chloracetamide	Chemo Puro Mfg. Corp.
Chemocide CETB	Cetyltrimethylammonium bromide	Chemo Puro Mfg. Corp.
Chemocide ETA	Ethylmercuri-thiosalicylic acid	Chemo Puro Mfg. Corp.
Chemocide HK	Heptyl *p*-hydroxybenzoate	Chemo Puro Mfg. Corp.
Chemocide LPL	Polychlorphenyl dodecanoate	Chemo Puro Mfg. Corp.
Chemocide PCMC	*p*-Chloro-*m*-cresol	Chemo Puro Mfg. Corp.
Chemocide PCPD	*p*-Chlorophenyl-propanediol	Chemo Puro Mfg. Corp.
Chemocide SA	Salicylanilide	Chemo Puro Mfg. Corp.
Chemocide XK	Hexyl *p*-hydroxybenzoate	Chemo Puro Mfg. Corp.
Dimethylamine borane		Callery Chem. Co.
Dodecyl boric acid		Callery Chem. Co.
Fungi-Ban	Bis(tributyl tin) oxide	Concor Chem. Co.
Merfenel 41	α-Acetoxy-mercuri-β-hydroxy ethane	R. T. Vanderbilt Co.
Merfenel 51	Phenylmercuric dimethyl dithiocarbamate	R. T. Vanderbilt Co.
Meta-san D-245	Phenylmercury salt complex of 2,4,5-trichlorophenol	Metalsalts Corp.
Meta-san D-246	Phenylmercury salt complex of 2,4,6-trichlorophenol	Metalsalts Corp.
Meta-san DM	Phenylmercury methacrylate complex	Metalsalts Corp.
Meta-san D-OH	Phenylmercury salt complex of phenol	Metalsalts Corp.
Meta-sol L	Diphenylmercury ammonium propionate	Metalsalts Corp.
Nalco 201	Blend of chlorinated phenols	National Aluminate Corp.
Nalco 236	Polyamine salt	National Aluminate Corp.
Nonyl boric acid		Callery Chem. Co.
Pyridine borane		Callery Chem. Co.
Quicksan C-10	3-Chloro-2-methoxypropyl mercuric acetate	Stecker Chemicals, Inc.
TBTO	Bis(tributyl tin) oxide	Metal & Thermit Corp.
Thienylmercuric acetate		Stecker Chemicals, Inc.
Tin Anti-Slime	Bis(tributyl tin) oxide	Amionic Chem. Co.
p-Toluenesulfonamide		Monsanto Chem. Co.
Trimethylamine borane		Callery Chem. Co.
Vancide 26 EC	Lauryl pyridinium salt of 5-chloro-2-mercaptobenzothiazole	R. T. Vanderbilt Co.
Vancide 51 Z	Zinc dimethyldithiocarbamate and zinc 2-mercaptobenzothiazole	R. T. Vanderbilt Co.
Vancide 89	*N*-Trichloromethylmercapto-4-cyclo-hexene-1,2-dicarboximide	R. T. Vanderbilt Co.

CAMPCO LOG BARKER



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Standard models are for logs 5"—36" diameter and lengths of 20', 24' and 26'. Special models available for West Coast hardwood and veneer logs up to 55" diameter and 30' lengths.

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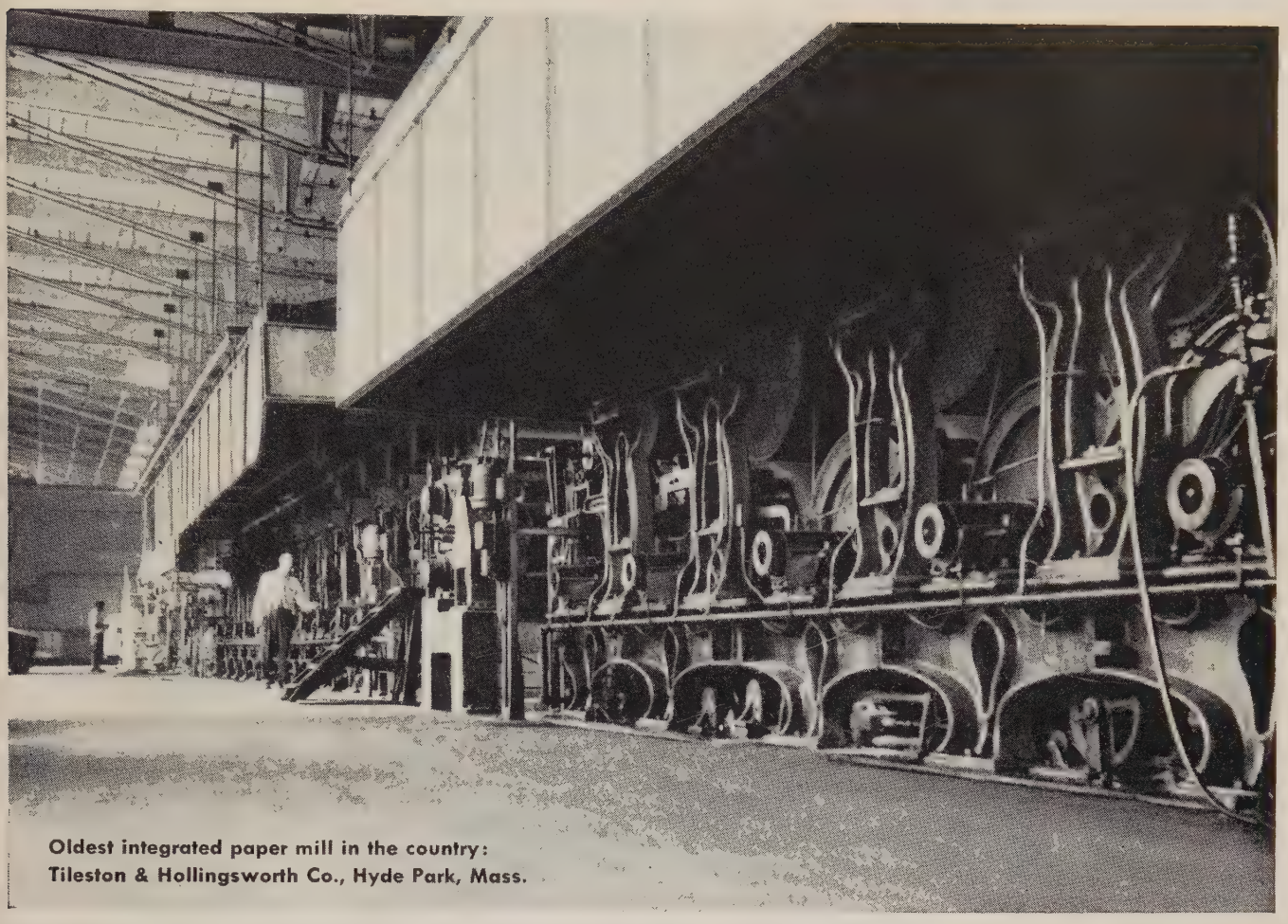
CARTHAGE MACHINE CO.
CARTHAGE, N. Y.



Oldest integrated paper mill in the country: Tileston & Hollingsworth Co., Hyde Park, Mass.

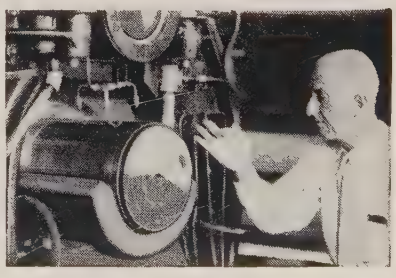
Lubrication engineer switches 230-year-old mill to Cities Service Lubricants

William D'Entremont, Lubrication Engineer for the Boston Office of Cities Service, spent three days at the Tileston & Hollingsworth paper mill making an extensive survey of their operations. He checked over dozens of machines with hundreds of lubrication points. His report convinced management to switch to Cities Service lubricants. Mr. D'Entremont's recommendations reduced the number of lubricants used by the company by one third, and at the same time increased efficiency of operations.

Typical of the Cities Service lubricants now used by Tileston & Hollingsworth are several types of Trojan grease, each fulfilling a specific and rigorous requirement. The unique properties of Trojan greases are the result of scientific use of laboratory-tested fortifying chemicals. Mineral oils comprise the largest percentage of the ingredients in Trojan greases. Only such oils are employed that are known to give the best results required of a given type—a knowledge gleaned from years of research and experience in the manufacture of high grade greases for a host of critical customers.

To learn how Cities Service lubricants can help you in your business, call the Cities Service Lubrication Engineer in your area. Or write: Cities Service Oil Company, Sixty Wall Tower, New York 5, N. Y.

The original charter establishing this company was issued in 1728 by the Provincial Government in Mass. In its first year the charter called for the company to make *"Two hundred Rheams of good Merchantable Brown Paper and Printing Paper"* plus *"Fifty Rheams of Writing Paper of equal goodness with the Paper commonly stampt with the London Armes."* Today the mill produces that much paper in an hour.



William D'Entremont checks paper mill's machinery. His recommendations reduced number of lubricants used by company by one third.

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Progress in Alkaline Pulping—1959

THE INSTITUTE OF PAPER CHEMISTRY[a]

This report continues the series (*1–13*) on the technical and topical literature reporting progress in alkaline pulping. It reviews the literature abstracted in the *Abstract Bulletin of The Institute of Paper Chemistry* during the calendar year 1959.

MODERNIZATION, EXPANSION, NEW MILLS

United States

Paper Mill News (*14*) tabulates the new mills, new machines, and expansion programs which have started operating in 1958 or are under construction or projected for 1959 and 1960. Evans (*15*) presents a survey of the pulp and paper industry during 1958 which includes a list of new mill and paper machines started up in 1958 and a list of major projects scheduled for completion in 1959 or later.

A description is given (*16*) of the background and recent expansions of the pulp and paper mills of Potlatch Forests, Inc., Lewiston, Idaho. The new kraft mill of the Boise Cascade Corp. at Wallula, Wash., is also described (*17*).

On the occasion of the mill's 75th anniversary, the Camas, Wash., mill of Crown Zellerbach Corp. is described (*18*). The growth of the mill is outlined, from its initial production of four tons daily to the recent installation of a Pandia continuous digester of 100 tons daily capacity.

Blackerby and Williamson (*19*) give a description of the new Georgia-Pacific Corp. kraft pulp and paper mill at Toledo, Ore. The chemical recovery and power plant systems of the new kraft pulp and paper mill of Dierks Forests Inc. at Pine Bluff, Ark., are described (*20*).

Owensby and Halsell (*21*) describe the installation, start-up experiences, and the results of operation of the Kamyr continuous digester at Gulf States Paper Corp., Demopolis, Ala. A detailed description (*22*) of the Brewton, Ala., pulp and paper mill of Container Corp. of America is given which covers wood-handling facilities, water-treatment plant, power plant, pulp mill, bleach plant, and paper mill.

Van Derveer and others (*23–27*) present descriptions of Marathon Southern Corp.'s pulp and paper mill at Naheola, Ala., producer of bleached kraft pulp, paper, board, towels, and tissue paper. Plans have already been announced for expanding the production capacity of the new mill by almost 50%.

Diehl (*28*) discusses the interchangeability of the two mills of Rayonier, Inc. at Jesup, Ga. They can operate as one unit or as separate units, one making paper pulp, the other dissolving pulp. A brief outline is given of the recently completed expansion and modernization program at the plant of Union Bag-Camp Paper Corp., Franklin, Va. (*29*). The new $20 million dissolving pulp and paper mill of Buckeye Cellulose Corp. at Foley, Fla., is described (*30*). A fully integrated water softening and lime recovery system produces the mill's entire lime requirement. The new Hudson Pulp & Paper Co. mill at Palatka, Fla., manufacturing tissue paper from southern kraft pulp is described (*31*).

Ray (*32*), Ludwig (*33*), and others (*34*) give descriptions of the new kraft pulp mill of the Eastern Fine Paper and Pulp Division, Standard Packaging Corp. (formerly Eastern Corp.), Lincoln, Me. The mill features the first Kamyr continuous digester installed primarily for hardwood pulping in North America. Several operating difficulties encountered and remedied are described in some detail.

Canada

A description is given (*35*) of the black liquor recovery and steam generation unit recently installed at the Kalamazoo Vegetable Parchment Co. mill, Espanola, Ont.

Blackerby (*36*) and Fitzsimmons (*37*) describe the recent $83 million expansion at the Port Alberni mill of MacMillan & Bloedel Ltd.'s Port Alberni Pulp & Paper Division, located on Vancouver Island, B. C. The mill is now producing groundwood and kraft pulp, paper, board, and newsprint.

Barclay and others (*38, 39*) discuss the progress of the construction of a new pulp mill for Celgar Co. Ltd. at Castlegar, B. C. Construction was to begin near March 1, 1958, and operations on or before March 1, 1961. It will be the first mill in British Columbia to install a Kamyr continuous digester system.

Europe

The annual survey of modernization and expansions in the Finnish forest products industries conducted by the Central Association of Finnish Woodworking Industries is presented (*40*). A description is given (*41*) of the history and present operations of Joutseno-Pulp Osakeyhito, Finland. The modernized kraft mill, put into operation in 1955, includes the use of Kamyr continuous digesters.

Whitcomb and others (*42, 43*) describe the Skogsagarnas Cellulosa AB. mill at Monsteras, Sweden. Scheduled for production in the fall of 1958, the mill will produce 75,000 tons/year of unbleached and semi-bleached kraft pulp, with an increase to 150,000 tons planned for the near future.

Svendsen (*44*) gives a description of the background and current operations of the Norwegian pulp and paper mill Saugbrugsforeningen at Halden. Brief mention is made that the mill will be the first in the world to use steam produced in a nuclear power plant. The plant is now being built.

Bertin-Roulleau (*45*) reports on the installation of a

[a] This survey for 1959 was prepared by J. R. Byrne, Research Assistant, Editorial Department, The Institute of Paper Chemistry, Appleton, Wis., under the supervision of Jack Weiner, Editor. Manuscript reviewed by Edward F. Thode, Research Associate, Engineering and Technology Section.

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RESYN 3600 is a water dispersed polyvinylidene chloride that promises a revolution in protective coatings. For the first time, it combines exceptional barrier properties with simple low cost application by high speed coating machines or spraying. It offers important new advantages:

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RESYN 3600 can be coated on paper, paperboard, plastic and other substrates. It also may be applied to fibers. It is of interest to a broad range of industries, including food and other types of packaging, converting, chemical, and textile.

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modern black liquor recovery plant at the Roquefort, France, kraft pulp and paper mill. Whitcomb (*46*) describes the recently completed pulp mill in southern France, Cellulose d'Aquitaine at St. Gaudens. The mill is the first prehydrolysis kraft mill in France; its capacity will be 100 tons per day of nitrating pulp or 140 tons per day of bleached paper grade pulp.

Kostrzewski (*47*) discusses some of the improvements made at the Krapkowice mill in the past five years. A description is given of the mill equipment and of the woodyard, digester room, black liquor recovery, and papermaking operations.

Bozidar (*48*) traces the history and development of the Zagreb Paper Mill, which produces mechanical and kraft pulps and a wide range of papers. Current expansions at the mill, to be completed in 1960, will more than double the prewar capacity of the mill.

Grindrod (*49*) reports that Billeruds AB. has recently put into operation an experimental pulp mill at its plant at Jossefors, Sweden. Probably the most essential innovation in the experimental mill is the cooking plant, which is equipped with a continuous two-stage Kamyr digester. The digester will be used for testing a number of processes so far not tried out on a major scale, including continuous two-stage sulfite and prehydrolysis sulfate cooking.

Formosa

Chen (*50*) describes the mill procedures and equipment of the Longlived Pulp and Paper Corp., Taipei, China, which was designed to produce bleached bamboo kraft pulp and an annual production of 3000 tons of various kinds of high-grade tissue papers. One of the objectives of the mill was to meet local demand for paper and reduce the cost of pulp and paper production.

Egypt

Following a brief review of the modern history of the Egyptian pulp and paper industry, now producing about 40% of the national consumption of paper and paperboard, Fouad (*51*) describes three new mill projects that are in the planning or construction stages

Australia—New Zealand

Cohen (*52*) gives a report on the pulp and papermaking developments in Australia and New Zealand. Recent innovations and plans described include a new tissue mill, a chlorine generating plant using radioactive mercury to control mercury losses, the partial substitution of cold soda eucalyptus pulp for eucalypt groundwood in newsprint, and the first completely enclosed hood in Australia. A description of the proposed expansion plans of New Zealand Forest Products Ltd. at the Kinleith mill is given (*53*).

ALKALINE PULPING—GENERAL

The kraft industry in the United States celebrated its 50th birthday in February (*54*). The first kraft pulp made in the United States was produced at the Roanoke Rapids Mfg. Co., Roanoke Rapids, N. C., on Feb. 26, 1909. A brief review of the development of the kraft process is given.

A review of the kraft pulping process by Chadeyron (*55*) covers the historical development of the process, the production of kraft pulp around the world, the chemical and mechanical properties of the pulp, and the chemistry and technology of the process.

Hoguet (*56*) presents a forecast of 1959 consumption figures and the reason behind each prediction for the various grades of pulp—unbleached kraft, bleached and semibleached kraft, groundwood, etc. Gains in demand are expected for every major grade of wood pulp.

Activities are reviewed and abstracts given (*57, 58*) of some of the papers presented at the 12th International Alkaline Pulping Conference of TAPPI, held in Hot Springs, Ark., Sept. 24–26, 1958.

A review of developments in the Canadian kraft pulping industry is given (*59*) which covers continuous cooking, bleaching, odor control, evaporation, recovery, and pulp washing and cleaning. Kraft mills in Canada are listed, and the methods or equipment used by each in relation to the fields covered are indicated.

Mihalache, and others (*60, 61*) give a review and preview of Romanian pulp and paper production. The technical and economic possibilities of introducing a high-yield sulfate pulping process at the Romanian "Reconstructia" pulp mill are discussed.

Watson (*62, 63*) and Reid (*64*) investigated the influence of wood storage on the pulping and papermaking properties of pine and eucalypt pulpwood. Kraft pulps were prepared from freshly cut wood and from samples which had been stored for varying lengths of time under various conditions. Results obtained indicate that it is best to pulp pine in the green condition, whereas underwater storage appears to be the least desirable procedure. Eucalyptus pulp showed no marked differences between green and airdry wood.

Nolan (*65*) claims that the use of airdry chips in laboratory kraft pulping studies has two advantages: the chips can be stored without spoiling and more accurate determinations of moisture content can be made. With these advantages in mind, the effect of pulping airdry wood was investigated. Dry chips showed lower screened yield and pulp quality, but rewetted chips gave rise to essentially the same pulps as did fresh chips treated under the same conditions.

Richter (*66*) obtained a patent for a method and apparatus for recharging a pulp digester in which the alkaline cooking liquor is introduced into the lower portion of a heated digester so that it forms a quiescent pool having a gradually rising level. This reduces liquor evaporation and digester corrosion.

Saarinen, *et al.* (*67*), found that sulfate pulping of birchwood to a yield of 59% gives a suitable animal fodder. This product gives a digestible raw carbohydrate yield of 51.5%.

THE COOKING PROCESS

Watson (*68*) pulped two wood species (pine and eucalyptus) by the kraft, soda, and sulfite processes in order to compare the effect of the type of cooking process on the properties of the pulps obtained. The results showed the expected grading for the various types of pulp from the two wood species.

Freedman (*69*) compared the two-stage soda and kraft pulping of northern hardwoods. Results indicate that not only are two-stage soda pulps stronger

The latest ways to make better white papers

news from Du Pont

The trend to high white papers is a fast growing one—and for many good reasons. Envelopes, letterheads, packages, printed pieces . . . all white items take on a little extra elegance, a little extra sales appeal when made from high-quality, sparkling white paper. And the graphic work, words and colors, usually has superior impact and visibility when set against a high white background.

See for yourself! This page is a high white paper, made with Du Pont Paper White SP Concentrated Solution. Compare this sheet with ordinary white papers, or even your "white" shirt. Quite a difference! And this is typical of the kind of results you can obtain from the whole family of Du Pont Paper Whites and brighteners.

There's a Du Pont product for any type of paper

Today, you can select from a wide variety of Du Pont products to improve the appearance and brightness of any white paper. There's a Du Pont dye or optical brightener to meet the needs of every grade and type.

Du Pont basic dyes are used for newsprint and other ground wood base papers. And the trend to brighter and brighter chemical pulps is leading to increased usage of Du Pont Paper White optical brighteners and "Halopont"® tinting colors, which actually produce the best results on the brightest pulps. Both "Halopont"® Blue RNM and "Halopont"® Brilliant Blue 2 RN give excellent results in combination with Du Pont's optical brighteners. Du Pont Paper Whites are widely used and give excellent results on many different grades of paper.

New Paper White SP solution works over wide pH range

Paper White SP can be helpful to certain mills handling special applications, such as surface sized grades. It contributes to easier production of a sized, filled high white paper, is adaptable to runs at various pH's, and has many other interesting characteristics.

- Can be used in either beater or surface applications.
- Shows relatively little graniting.
- Can be used in the beater at the pH desired for maximum filler retention.

(Continued on back page)

• Is especially good for surface applications at a low pH.

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For most white papers, "Halopont"® tinting colors are recommended. These economical colors provide good lightfastness as well as brightness.

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Du Pont Color Council Developing "White Paper Criteria"

To aid manufacturers, buyers and specifiers of paper further, the Du Pont Color Council is developing techniques and materials to help make the selection of white papers easier. A natural companion to the "Color on Colored Paper Criteria," the projected "White Paper Criteria" will assist the paper seller and buyer to create, predict and control graphic effects on different "colors" and textures of white papers more accurately. White will be one of the colors demonstrated in advertising and promotional literature produced by the Council as well.

For further information on how the Du Pont Color Council is helping the paper industry and trade to make and sell better papers more profitably, see your Du Pont Dyes and Chemicals representative.

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Dye Leveling	Du Pont Retarder LAN
Felt Washing	"Alkanol"® HCS surface active agent "Duponol"® RA surface active agent
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Linting Reduction on Printing Grades	"Zoron"™ WS resin finish
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Starch Insolubilization	"Zoron"™ WS resin finish
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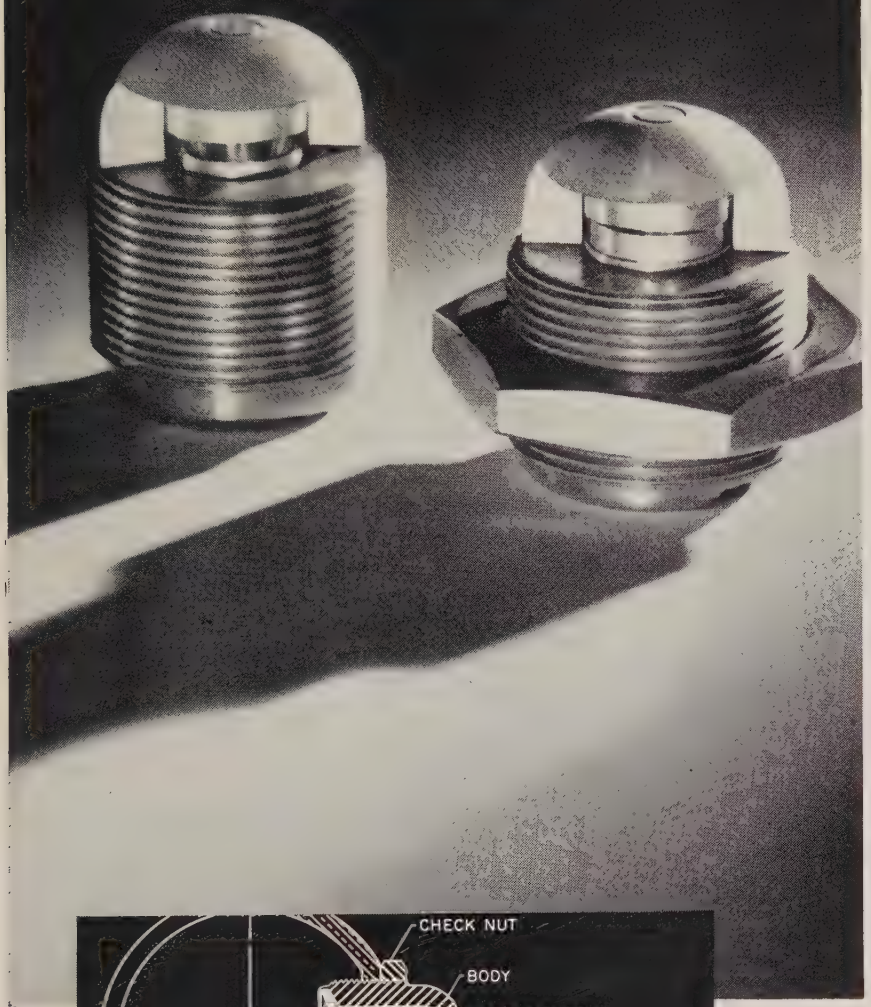

In mill after mill Lodding Showers are conserving water — cutting operating costs — performing effectively.

In showers it's the nozzle that counts. And Lodding Nozzles have been engineered to give you the greatest possible showering efficiency, whether your system uses fresh or clarified white water.

Lodding Nozzles are designed with a built-in, sealed piston. In operation, pressure of the water forces the piston against the spray button, which is precision-milled to create the exact spray pattern desired — from an 80° fan to a needle stream.

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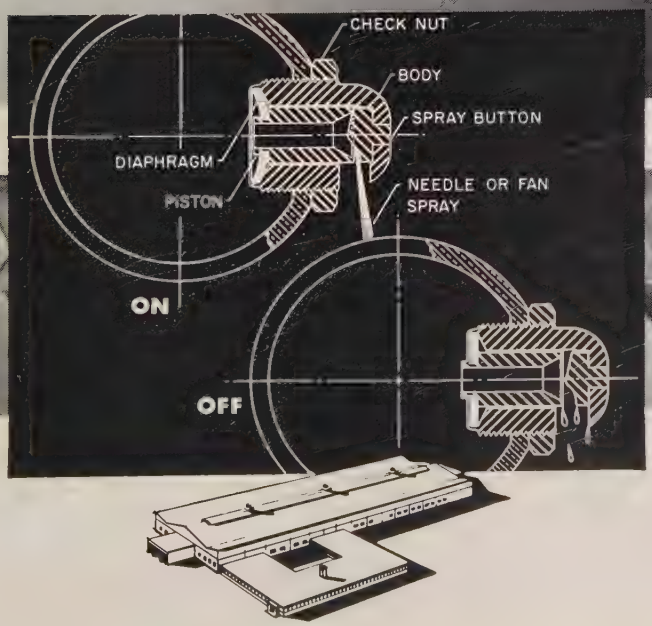

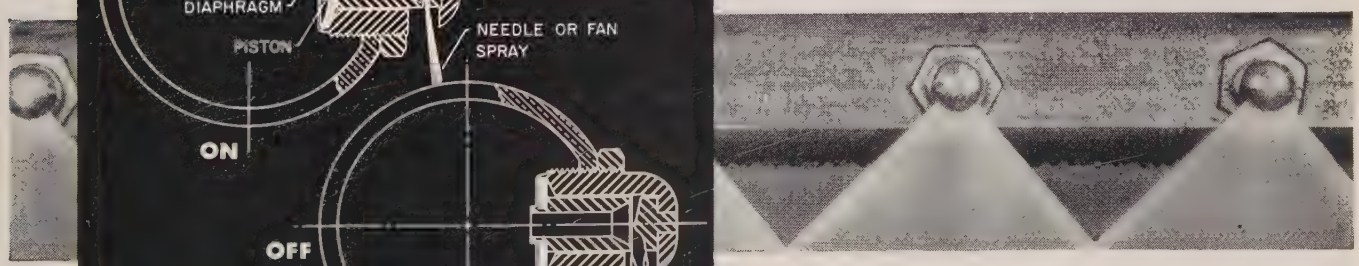

LODDING
Engineering Corporation
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than conventional soda pulps but they are as strong as kraft pulps, and even surpass them in some instances.

Stockman and Sundkvist (*70*) investigated the high-temperature kraft pulping of pine chips. Yield and chemical consumption decreased with increasing temperature. Pulp bleachability and strength properties were almost unchanged up to 200°C. Although cooking time was considerably shortened, this advantage can only be used in continuous pulping.

A patent by Rogers, *et al.* (*71*), concerns a process for producing a dissolving pulp for use in viscose rayon in which wood chips are subjected to an acid prehydrolysis, pulped in alkaline liquor, and bleached by a multistage process. Kosaya (*72*) carried out laboratory experiments to determine the effect of process variables on the quality of dissolving-grade kraft pulp from prehydrolyzed Siberian pine and larchwood.

Colton, *et al.* (*73*), obtained a patent for a kraft pulping process which consists of cooking the wood in the presence of potassium sulfide polymers at a chemical-to-wood ratio of 4:1 and at a temperature of about 160 to 180°C.

Litvinova, *et al.* (*74*), carried out laboratory cooks using sulfite liquor in the first stage and a modified kraft liquor in the second. High alpha-cellulose content pulp was obtained in very good yields. Pulps manufactured by this process could be bleached by the usual multistage process. Wenzl (*75*) received a patent for a multistage alkaline pulping process which comprises impregnating the raw material with an alkaline solution of 100 g.p.l. active alkali at 100°C., draining off the excess solution, heating the material to 140 to 180°C., displacing the alkaline solution with one of about half the concentration of the original liquor, extracting the noncellulosic material with this liquor, reducing the temperature, displacing the extraction liquor with dilute alkaline solution, and finally washing the pulp produced. A dissolving pulp of high alpha content is obtained.

Nepenin and Vybornova (*76*) prepared kraft pulps by substituting potassium for sodium in the preparation of the cooking liquors. In all experiments the substitution accelerated the digestion process. Unbleached potassium-base pulps had alpha-cellulose contents higher by 2 to 4% than its corresponding sodium-base pulps, were beaten more easily, and were characterized by improved mechanical strength properties.

Sobolev and Schuerch (*77*) discuss pulping wood with sodium hydroxide liquor which has been hydrogenated in the presence of nickel catalyst. The pulp obtained resembles soda pulp but is of a lighter color. A number of experimental pulps are described in detail.

Horio and Takahama (*78*) report that separating the prehydrolysis step from the actual cooking process in making kraft dissolving pulp and converting the prehydrolysis step into a continuous operation gives some very satisfactory results. The kraft cooking time was shortened 50% and some digester corrosion problems were eliminated.

Obenshain (*79*) obtained a Canadian patent for his process of continuous pulping which utilizes a liquor having an input concentration of 20 g.p.l. or less of sodium oxide which is circulated through the digester at 10 to 100 times the rate of chip flow. Richter (*80*) reports on the development of the continuous pulping of kraft. A steadily increasing tonnage is being produced all over the world with the annual production of Kamyr pulp at approximately 1.5 million tons. Meinhold and Knight (*81*) describe the Kamyr system used for the kraft pulping of hardwoods at the Eastern Corp. plant at Lincoln, Me. Tucker (*82*) outlines experiences in the operation of the 300 tons per day Kamyr digester at the Demopolis pulp mill of Gulf States Paper Corp.

Jain (*83*) reviewed the chemical mechanisms involved in several of the major chemical pulping processes, including kraft.

Kottek (*84*, *85*) studied the reaction between sodium hydrosulfide and a representative sample of *Eucalyptus regnans* at a series of pH levels. During cooking the pH controlled the amount of lignin dissolved, even after correction had been made for the delignification caused by the buffer solution. Wood cooked with sodium hydroxide after it had been treated with sodium hydrosulfide gave a greater yield and lower pentosan and cellulose losses for any lignin content than did cooking without pretreatment.

Karlivan and Odintsov (*86*) investigated the chemical changes which take place in lignin during soda and kraft cooks. Black liquor samples were taken throughout the cooking process and analyzed.

Krajcinovic, *et al.* (*87*), determined the degree of delignification during the kraft process using several different cooking liquors and found that the best results were obtained at 40% total sulfidity with the addition of polysulfides in an amount corresponding to about 12% sulfidity. Ishizu, *et al.* (*88*), found that during the soda or sulfate cook, ligninunder goes both an alkaline cleavage of its ether groups and a simultaneous condensation reaction. The study by Matsugu and Shiraiwa (*89*) shows that the reactivity of softwood lignin decreases during the initial stage of kraft cooks while the reactivity of hardwood lignin remains constant.

Nolan (*90*) presents evidence to show that delignification in kraft pulping is accomplished through the penetration of a reaction interface rather than penetration of the liquor into the chip, followed by the pulping reaction. The evidence is based on the physical appearance of partially cooked chips, the lignin gradient measured across partially cooked blocks, cooking curves, and the analyses of pulps and black liquors resulting from pulping under widely divergent batch conditions.

Fahmy and Fadl (*91*) found that in the alkaline pulping of wheat and rice straws, the duration of the cook exerted a greater influence on the solubility of mineral matter than did any of the other cooking variables.

Sato and Shimoda (*92*) cooked two species of wood by the kraft process using a constant alkali:wood ratio and varying the sulfidity of the white liquor from 0 to 100%. It was concluded that 30 to 50% sulfidity is optimum for shortening the cooking time and for improving the properties of kraft pulp. Il'inskaya (*93*) prepared alkaline cooks while varying the sulfidity from 0 to 75%. To obtain pulps of high D.P. and good mechanical strength, suitable for the manu-

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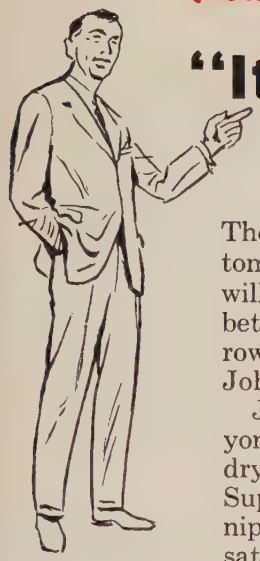

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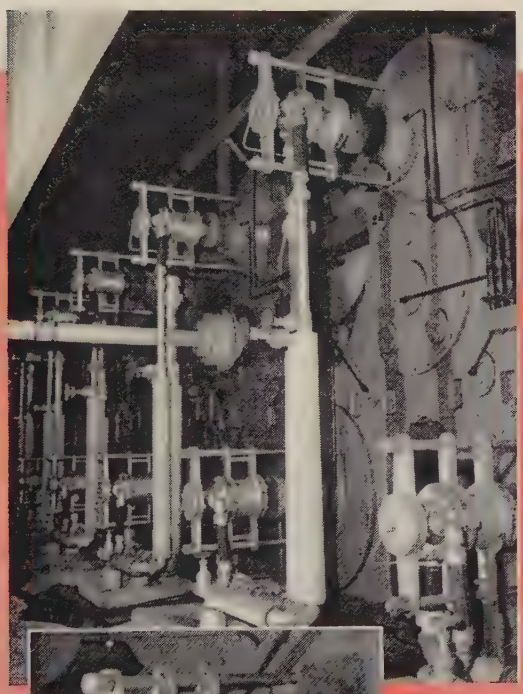

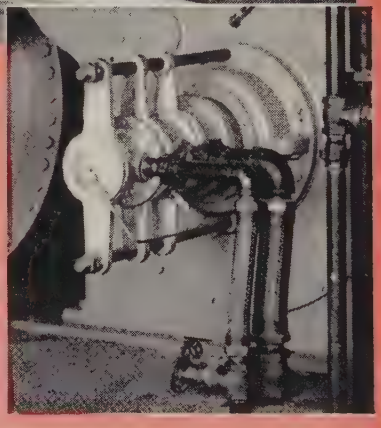

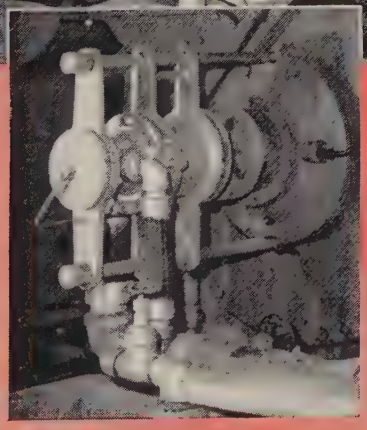

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facture of electrical insulation paper, the sulfidity of the liquor must be maintained at a level of 30 to 35%.

Owen (*94*) reports that a series of mill trials on cooking with elemental sulfur showed that it is entirely satisfactory as a means of adjusting sulfidity. No sustainable objections to addition at digesters were found.

Kerr and Hart (*95, 96*) investigated the properties of spruce kraft pulp obtained throughout the cooking cycle in a range of yields from approximately 45 to 90%. Both the unbleached and bleached pulps developed burst strengths which went through a maximum at about 51% yield.

Alhojarvi (*97*) discusses the supply, composition, and utilization of birchwood, in particular its holocellulose, in Finland with special reference to the kraft and other pulping processes.

Baczynska (*98*) investigated the suitability of willow as a raw material for the pulp industry and found that the mechanical strength properties of willow pulps are inferior to those of pinewood and poplarwood pulps and comparable to those of reed pulps. Vámos (*99*) surveyed the available literature on the utilization of willow and found that often contradictory results were obtained. It can be concluded, in general, that these pulps can be utilized only for the manufacture of low-quality papers and fiberboards.

A report of the U. S. Forest Service (*100*) states that hardwood timber resources in the Lake States region are available in sufficient quantity to support a substantial expansion of the pulp and paper industry for newsprint, hardwood kraft, and cold soda pulp. Perkins (*101*) reports on a project set up to evaluate the principal hardwood species available to both the Jacksonville and Pensacola, Fla., mills of St. Regis Paper Co. From the tests, magnolia, water oak, black gum, and red gum appeared to be the most desirable species. Overbeck and Agottani (*102*) prepared a series of kraft cooks from 11 species of Brazilian hardwoods. All but two species proved satisfactory. Each species in the mixture reacted as it did when pulped alone; hence, pulp uniformity could not be improved by varying the cooking conditions. Sedivy and Svaton (*103*) discuss the composition of newsprint stock and the mechanical properties of newsprint made from some of the new materials being utilized to replace softwood sulfite and mechanical pulps which include semichemical pulps made by the semikraft and cold soda processes and kraft pulps from hardwoods.

Skalicky and Vondrakova (*104*) report that satisfactory printing and parchment papers were produced from pulp blends containing 10 to 50% kraft beech pulp. The addition of beechwood kraft increased the opacity of the papers, but lowered their breaking length and folding endurance. The addition of 20 to 30% beechwood kraft is recommended for the manufacture of parchment paper. Gajdos (*105*) studied the problem of optimum prehydrolysis in the manufacture of beech kraft and came up with the following conclusions: For the manufacture of inferior quality viscose, prehydrolysis of chips at 150°C. for 150 min. can be recommended; if high quality viscose is desired, the chips must be prehydrolyzed for 4 hr. at 150°C.

Fedorishcheva (*106*) suggests that Daur larch be utilized for the production of alcohol and for the manufacture of unbleached and bleached kraft pulps and fiberboards. Zaĭtseva (*107*) recommends a procedure for the alkaline cooking of extracted larchwood chips which consists of cooking the chips for two hours at a maximum temperature of 173°C. with a white liquor containing 28% active alkali and having a sulfidity of 27%. In further studies by Zaĭtseva and co-workers (*108*) it was found that varying the sulfidity of the white liquor used in cooking larch affected the yield and hardness of the pulps, but not their mechanical properties. In trials using both extracted and unextracted chips, yield and hardness increased with increasing sulfidity.

In order to evaluate the utility of Colombian hardwoods, Chidester and Schafer (*109*) pulped 31 samples in a single mixture and in three selected mixtures. All pulps prepared exhibited sufficiently satisfactory properties to permit utilization in one or more of the paper product classifications. The newsprint experiments in particular were satisfactory.

Kozmal and Bucko (*110*) investigated acaciawood as to its possible utilization in the pulp and paper industry. Kraft pulps were obtained in a yield of 38 to 41% and demonstrated good mechanical strength properties. Acaciawood pulps can find applications in the manufacture of absorbent papers and other products for which a material of high specific volume is desirable.

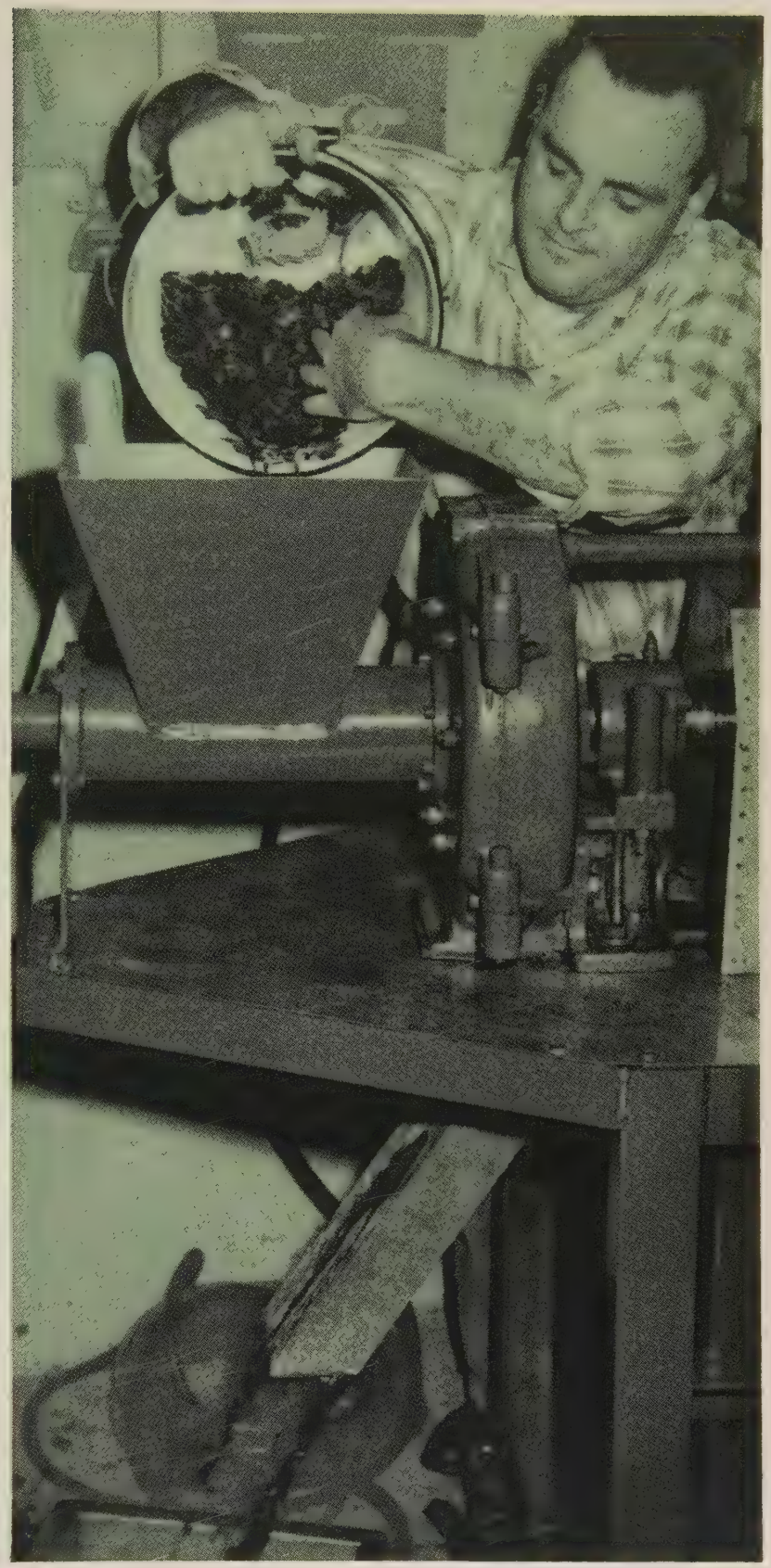
Fahmy and Ashmawy (*111*) found that *Eucalyptus camuldulensis* could not be pulped adequately by the usual sulfite process if a good viscose pulp was desired. Further investigation showed that a prehydrolysis kraft or soda cook, followed by hot refining, would produce a suitable viscose pulp.

Skrigan, *et al.* (*112*), studied the chemical properties and possible utilization of kraft pulps prepared from logging waste, sawmill waste, peat wood, and flax waste. It was concluded that the wastes could be utilized for the manufacture of pulps, board, paper, and insulating building panels. Phillips and Watson (*113, 114*) reported on investigations of the pulping and papermaking properties of New Guinea woods. Kraft pulps with good strength properties were prepared from *Araucaria* veneer waste, using very short cooking times and temperatures as low as 155°C. The pulp can also be bleached satisfactorily by conventional methods.

Kuniak, *et al.* (*115*), report that the depithed stems of cirok, recently introduced to Czechoslovakia, can be pulped with good success by the alkaline process. The pulps are bleached easily by the conventional three-stage process and can be used for the manufacture of quality white papers.

Pessolano (*116*) presents a long, general discussion on bagasse as a raw material for pulp and paper. Cusi (*117*) describes the basic methods employed in pulping bagasse at the San Cristobal mill. A description is also given of the structure of bagasse and the three fractions of principal importance in the pulping operation: the pith or parenchyma, the central vascular bundles, and the rind vascular bundles. Mitra (*118*) reports that viscose-grade dissolving pulps have been produced with relative ease from depithed sugar-cane bagasse by the sulfate process, but they were all high in pentosan content. A two-stage process involving acid prehydrolysis and sulfate digestion was highly effective in the removal of pentosans to the desired

SOLVAY "MINIATURE MILL" SCALES DOWN A CUSTOMER'S PULPING PROBLEMS



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low level. The commercial feasibility of the process is discussed.

Knapp and Wethern (*119*) obtained a patent for a rapid kraft process for pulping bagasse, straw, and other nonwoody materials so that no subsequent mechanical defiberization is necessary. It consists of mixing the material with a liquor containing sodium hydroxide and sodium sulfide, heating the mixture under pressure, and then maintaining the pressure and temperature for up to 5 min.

Ernst, *et al.* (*120*), carried out an experiment to study the 15-minute pulping of wheat straw at caustic concentrations of 4 to 10% and temperatures of 100 to 180°C. Data were obtained on regular handsheets and on nine-point corrugating medium produced in the laboratory. Fahmy and Fadl (*121*) prepared viscose pulps from wheat straw by a prehydrolysis-soda process. Krach and Lang (*122*) carried out laboratory kraft and semichemical cooks using *Melitotus officinalis* straw, rye straw, reed, and mixtures of the three. Mixed kraft pulps had strength properties comparable to those of pure rye straw or reed pulps.

Chang (*123*) describes the bamboo pulping and pulp bleaching operations employed at Longlived Pulp & Paper Corp., Ltd., Taiwan, in preparing stock for use in the manufacture of bond papers and tissues. Bhargava, *et al.* (*124*), found that impregnating bamboo chips with hot kraft white liquor resulted in a reduced cooking cycle and in pulp yields higher than those obtainable by conventional commercial pulping procedures. The pulps could be utilized in the manufacture of various printing and writing papers. Karnik (*125*) describes laboratory experiments on the preparation of high alpha-cellulose pulps from bamboo by a water prehydrolysis-sulfate process. Pulp was obtained, after bleaching, in a yield of 27.3% with an alpha-cellulose content as high as 93%.

Bakk (*126*) reports that the size of reed and straw chips has no decisive effect on either the cooking rate or the pulp quality obtained in alkaline digestions. However, the use of 20 to 30 mm. is recommended because it increases the charging capacity of the digesters.

Derbentsev and Kheifets-Polisskaya (*127*, *128*) pulped reed by the kraft process and by the sulfite-sulfate process. The best conditions for kraft cooking were found to be: sulfidity 20%, concentration of white liquor corresponding to 20% total chemicals, and a cooking time of 45 min. at 180°C. Under these conditions the yield of unbleached pulp is 47 to 48% and its hardness 55 to 60 permanganate units. The sulfite-sulfate cooking of reed gives pulps of higher yield and better bleachability. The sulfite-sulfate pulps contain more ash and lignin, but their mechanical properties are superior to those of kraft pulps.

Ikhel'zon (*129*) pulped reed growing in the Volga river delta by the soda, kraft, sulfite, and sulfite-sulfate processes. The two last processes seemed to be the most promising. Fahmy and Abou State (*130*) prepared suitable viscose pulps from Egyptian reed by the prehydrolysis-sulfate process. Yarn spun from two reed pulp viscoses compared favorably in mechanical and chemical properties with commercial wood pulp rayon.

Karnik (*131*) prepared satisfactory soda and kraft pulps from a mixture of *Cymbopogon martini* and *Sehima nervosum* grasses. When mixed with bamboo pulp, this pulp could be used in the manufacture of printing paper. Khristov (*132*) converted three species of grass (*Phalaris bulbosa*, *Panicum virgatum*, and *Elymus arenarius*) by the kraft process into pulp suitable for the manufacture of paper. The high lignin content of the grasses causes no technical difficulties when they are pulped in mixtures with wheat straw.

THE LIQUOR CYCLE

Jönsson (*133*) discusses the recovery of liquor through pressing. With kraft pulp, three or more presses in series are required to achieve results comparable with those obtained in diffusor washing. Some of the advantages of presses are moderate power and space requirements, closed system operation, absence of foaming, and good recovery yields. Disadvantages include plugging, risk of deposits on screen plates, and high investment costs for series installations.

Gelo (*134*) reports on a potentiometric determination for sodium sulfide in black liquors which gives an average percentage error lower than that for the TAPPI titration method. However, the values were higher by 7% than those obtained by the TAPPI method. The entire determination takes only 30 min. and is particularly suitable for routine tests in the mill. Bilberg (*135*) states that potentiometric methods for determining sulfite and thiosulfate have been developed for the analysis of oxidized black liquor under nitrogen.

Prey and Kabil (*136*) found up to 0.52% lactic acid in beechwood black liquors and up to 0.26% in sprucewood black liquors. The determinations were based on paper-chromatographic procedures.

Adams and Koppe (*137*) present a gas-chromatographic technique for the separation and identification of complex mixtures of volatile sulfur-containing compounds of the type found in kraft pulp digester blow gas and black liquor combustion products. A new series of nine column solvents of the alkyl aryl polyether alcohol type is proposed.

Gelo and Wasiak (*138*) developed a method for determining sodium sulfide, sodium hydroxide, and sodium carbonate in black liquor. The method was found to give results of satisfactory accuracy and reproducibility. The relatively simple determination can be carried out in 45 min.

Dyck (*139*) describes the new soda ash recovery system developed by Western Precipitation Corp. The process involves converting the sulfides and sulfites to thiosulfates in an oxidizer and then concentrating the resultant liquor in standard evaporators. Kubelka and Votoupal (*140*) present a review of the kraft recovery system and discuss the chemical reactions which take place during the various stages of the process. Thomsen (*141*) obtained a patent for a method of regenerating spent pulping liquor which can be applied to the kraft, soda, and sulfite processes.

The Chesapeake Corp. of Virginia at West Point has put into operation its third black liquor recovery unit, a 500-ton boiler (*142*). Venemark (*143–145*) continued his series on black liquor evaporation with parts 2 through 4. The subjects covered are: volatile acids of black liquor, degassing and hydrogen sulfide recovery, and corrosion of carbon steel by black-liquor vapors.

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Jacoby and Lankenau (*146*) describe some typical cases of evaporator corrosion and point out the relationship to the mill liquor system. Sources of corrosion include normal liquor, boilout liquor, improper materials of construction, and mechanical design.

Patents by Flynt and Suda (*147, 148*) describe a method for concentrating spent liquors which consists of bringing the liquors from the pulp washers into direct contact with the hot flue gases from the furnace. The method is most suitable for use by smaller pulp mills.

Hauck and Suda (*149*) obtained a patent for a combustion furnace which includes two combustion chambers, one for burning black liquor, the other for burning oil or gas fuel. The furnace is connected with a single steam-generating and superheating unit.

Moonan (*150*) obtained a Canadian patent for an apparatus in which concentrated black liquor is reinforced with the desired chemicals and mixed sufficiently so that the solids are broken up enough to allow them to pass through the spray nozzles of the recovery furnace. Moxness (*151*) patented an apparatus in which the liquor is fortified and then sprayed into the furnace.

Thurmond and Bristow (*152*) describe the installation of a wire mesh separator to reduce entrainment in a multiple-effect evaporator. Tests showed that black liquor loss in the evaporator had been practically eliminated. The separator can also be applied to dissolving tank vents. Blessing (*153*) reports on the operation of the Research-Cottrell wet-bottom electrostatic precipitator at the Longview Fibre Co. in Washington. The precipitator recovers about 38 tons of salt cake per day from the gases produced in the recovery furnace which burns 2,000,000 lb. daily of black liquor solids.

Boyer (*154*) enumerates the three principles which must be considered when integrating kraft recovery with NSSC production: green liquor phase separation, the 50% sulfidity rule, and the desulfonation reaction with sulfite liquor.

Martin (*155*) describes a process for the introduction of elemental sulfur to the kraft liquor system at the point where the black liquor leaves the bottom of the electrostatic precipitator.

A patent by Knight and Varcoe (*156*) describes a clarification tank which is combined with storage and surge facilities in one unit to insure a constant liquor flow regardless of variations in the supply of green liquor entering the unit.

Alcock and Saul (*157*) report on the development of a vertical mobile cathode cell for the electrolysis of soda-process green liquor. The electrochemical process is an alternative to the lime causticization of green liquor, and therefore, the undesirable features of causticization and lime-kiln operation are eliminated.

Sylwan (*158, 159*) presents some experiences concerning the optimum operating conditions in the causticizing plant of a sulfate mill and discusses the conditions for the complete utilization of the lime and for complete chemical recovery.

Bristow, *et al.* (*160*), report that considerable laboratory and mill experimentation at the Savannah mill of Union Bag-Camp Paper Corp. has resulted in a hydraulic salt cake distributing system which is cleaner, easier to operate, capable of closer control, and less demanding of maintenance than the mechanical system it replaced.

Boyer (*161, 162*) obtained patents for a method for separating sodium sulfide and sodium carbonate from each other and from a smelt containing the two. The liquid smelt from the furnace is subdivided so that it solidifies in finely divided form and then dissolves in water. The sodium carbonate is then crystallized out.

Laboureur (*163*) obtained a patent for a process of purifying and utilizing black liquor. The organic material is removed and the remaining matter is treated with a fermentation agent. The fermentation products are of use as fertilizers, cattle feed, and soil conditioners. Shelow and Pickens (*164*) describe a modification on the standard type of sulfate soap collection system which has resulted in a recovery of up to 175 lb. per ton of pine pulp.

Witek (*165*) outlines a program for the utilization of sulfate turpentine in Poland which is now wasted, and also gives a brief description of the chemical processes used for the synthesis of some compounds which can be made from sulfate turpentine.

STREAM AND AIR POLLUTION

Bialkowsky and Brown (*166*) present a review of pollution control as practiced in the pulp and paper industry. The pulping processes are described and methods for disposing of the spent liquor and recovering chemicals therefrom are discussed.

A description is given (*167*) of the construction of an 1800-ft. extension onto the plant-to-ocean pipeline for waste disposal at the Toledo mill of Georgia-Pacific Paper Co. A previous 1100-ft. extension proved insufficient to prevent the waste from being washed up on the beach.

Bulicek (*168*) gives a review of the water pollution problem in Czechoslovakia and comments that, although kraft mill effluents constitute a small fraction of total mill effluents, their effective purification is equally important because of the high toxicity of their components and the odor problems.

Berger and Brown (*169*) describe experiments in which caustic extract from a bleached kraft mill is treated by application to a precoat of hydrated lime on a rotary vacuum filter. The lime-lignin reaction takes place on the surface of the precoat and can be doctored off and fed to the kiln along with lime mud. Color removal is in excess of 95%.

Tötterman (*170*) discusses various ways of treating mill effluents for minimization of stream pollution in the pulp, paper, and board industries. Crawford (*171*) reports that a spray irrigation system for disposing of certain mill effluents was included in the recent expansion of the Union Bag-Camp Paper Corp. kraft mill at Franklin. The new system has resulted in an 86% removal of the total B.O.D. and suspended solids load in the total mill effluent.

Coleman (*172*) reports that the burning of relief and blow gases in the lime kiln has been proved practical from an operational standpoint. Odor elimination has improved because there is less down time than with the separate gas-burning furnace.

Zielinski (*173*) reviews the formation of volatile sulfur compounds during the kraft process, their distribution in flue gases and fumes, and methods used for

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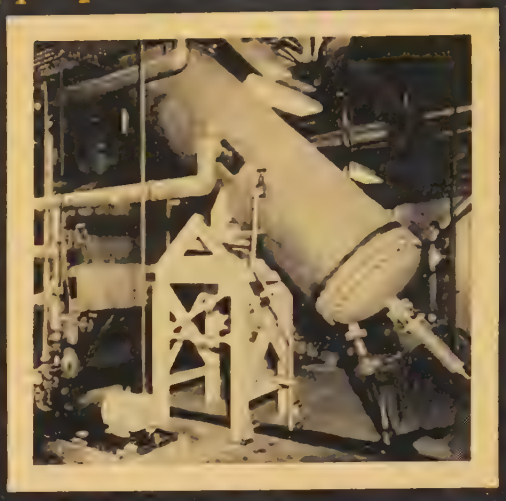
Bauer pulping and refining equipment in Blandin chemi-mechanical pulp mill



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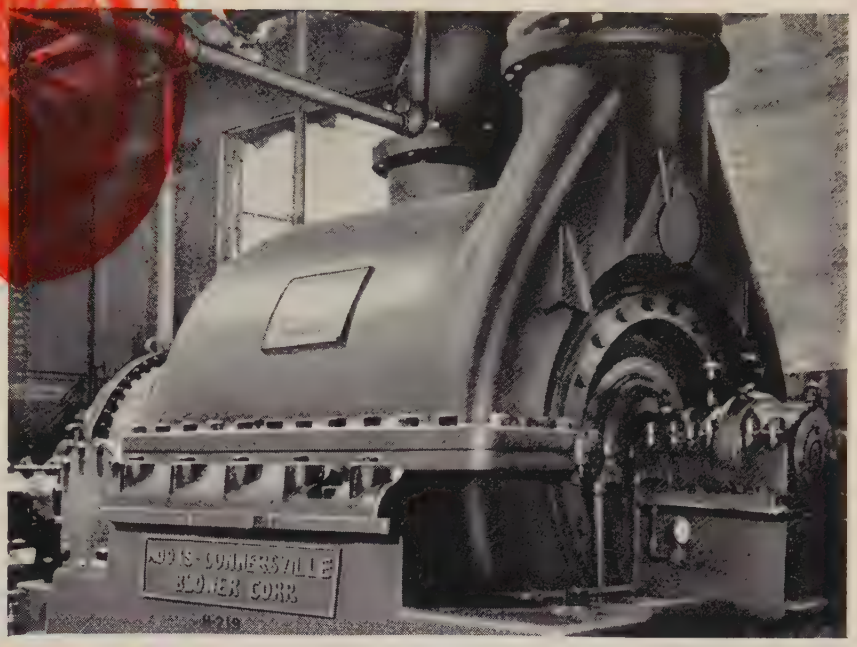
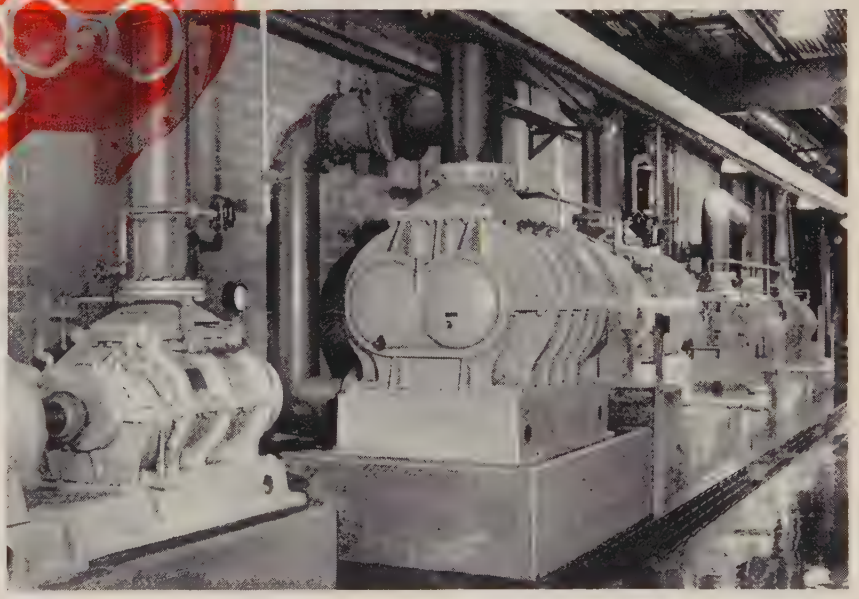


Roots Rotary Pumps supplying vacuum on large paper machine in Texas mill.



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their elimination. The possible industrial utilization of these compounds is discussed and methods for recovering them are mentioned.

Limerick (*174*) obtained a patent for a method for reducing kraft mill odors which consists of mixing the stack gases with 10–100 p.p.m. ozone. Maksimov (*175*) reports that a system for odor abatement by oxidation of black liquor and noncondensing gases was installed at the Svetogorsk Combine. Preliminary tests indicated that the system is suitable for the simultaneous purification of gases from digesters, washers, and evaporators.

Lardieri (*176*) discusses air pollution control and its relation to the pulp and paper industry. He states that the kraft industry does not seem to have a critical air pollution problem at this time. Various recovery processes are discussed and capital costs of the equipment are compared.

Hendrickson (*177*) reviews the contaminants which may contribute to air pollution in sulfate pulping and describes equipment used for sampling the air as well as the analytical procedures for testing the samples.

Trobeck, *et al.* (*178, 179*), describe the odor control measures employed at the kraft pulp mill Fabricas de Papel Loreto y Pena Pobre, S.A., Mexico. Odors from the evaporators are eliminated by oxidation of the black liquor. Odors from other sources are drastically reduced by using the reaction between chlorine and gaseous sulfur compounds.

BLEACHING

A description is given (*180*) of the seven-stage bleaching system used to bleach kraft pulp at the pulp mill of the Royal Swedish State Forest Industries, Ltd. at Karlsborg. Grindrod (*181*) reports that the plant has a capacity of 60,000 tons per year of bleached kraft pulp.

Sato (*182*) reports on a five-stage bleaching process which was used on kraft beechwood pulps. It was found that sodium carbonate could be used with considerable cost savings in place of sodium hydroxide for alkaline extraction after chlorination.

Colombo, *et al.* (*183*), studied the effect of various bleaching stages and agents on the mechanical properties of kraft pulp. Voelker and May (*184*) report that drying prior to bleaching had no adverse effects on the bleaching characteristics of pine kraft; however, it impaired the bursting strength and tensile strength properties of the pulp both before and after it was bleached.

Monzie, *et al.* (*185*), report that experimental data indicate that the yellowing of kraft pulps is related directly to the residual hemicellulose content. At equal degrees of oxidation, cellulose has a stronger tendency to color reversion than hemicellulose.

Outterson, *et al.* (*186*), obtained a patent for a process for both bleaching and sizing kraft pulp in a unitary operation. A slurry of pulp, hypochlorite bleach, and rosin size is prepared and the hypochlorite allowed to react with the pulp. Alum is added after 0.5 hour and it reacts with the residual hypochlorite and precipitates the rosin size.

Algar, *et al.* (*187*), describe the bleaching process being used at the Maryvale mill of Australian Paper Manufacturers, Ltd. The bleached pulp is made by a two-stage batch process using a combined calcium hypochlorite-chlorine gas first stage and a combined sodium hydroxide-calcium hypochlorite second stage. The main advantage of the process lies in increased production rate from a batch plant when making pulps of approximately 80% brightness.

Zaĭtseva (*188*) reports that hypochlorite bleaching of larchwood kraft pulp from extracted chips yields bleached pulps having an alpha-cellulose content of 94 to 95%, even without hot alkaline refining. Paulson (*189*) claims that the reaction between sulfate pulp and hypochlorite may be simplified by treating it as a cellulose depolymerization reaction in which the lignin and hemicellulose fractions compete for the hypochlorite.

Jensen, *et al.* (*190*), report that replacing one or both of the hypochlorite stages in the final bleaching of birchwood kraft with chlorine dioxide resulted in higher brightness and strength properties. A description is given (*191*) of the bleaching system of the Penobscot Chemical Fibre Co., Maine. They are bleaching both soda and sulfite pulps by chlorine dioxide stages.

Ferguson and MacDonald (*192*) describe the design and operation of a new bleach plant for producing high-brightness soda pulp at the Cornwall, Ont., mill of Howard Smith Paper Mills, Ltd. A new chlorine dioxide generator using a modified Mathieson process was also put into operation.

Chadwick, *et al.* (*193*), have concluded that the best bleaching process for kraft pulp consists of the following five stages: chlorination, alkali extraction, hypochlorite, chlorine dioxide, and peroxide (CEHDP). This sequence gives a G.E. brightness of 89 to 91%. Strength of the pulp is good and brightness stability outstanding.

A bleaching process patented by Schroeder (*194*) consists of bleaching the pulp with chlorine, extracting with alkali, bleaching the extracted pulp with alkaline hypochlorite, acidifying the pulp with a mineral acid to a pH of 4.0 to 5.5, and subjecting the acidified pulp to at least one alkaline peroxide bleach. Lindahl, *et al.* (*195*), present a technical and economic comparison between peroxide and chlorine dioxide bleaching.

Stalter and Chadwick (*196*) report that a three-hour aging test at 85°C. and 100% R.H. has proved satisfactory for determining the relative brightness stability of bleached pulps.

CORROSION

Kesler (*197*) presents the supplement to the bibliography of alkaline digester corrosion which consists of 12 annotated references from 1956 to 1958.

Canavan (*198*) reports on the current status of alkaline digester corrosion as summarized from data collected from 30 mills covering 229 digesters. The average corrosion rate for carbon steel digesters has decreased from 28 mils to 21.5 mils per year. The critical area still appears to be confined to the middle shell, and liquor charging is still leading the list of causes for corrosion in the critical area. A report by Canavan (*199*) three months after the preceding one showed that the average corrosion rate increased from 21.5 to 22 mils per year. The critical area appeared to have moved to the upper shell.

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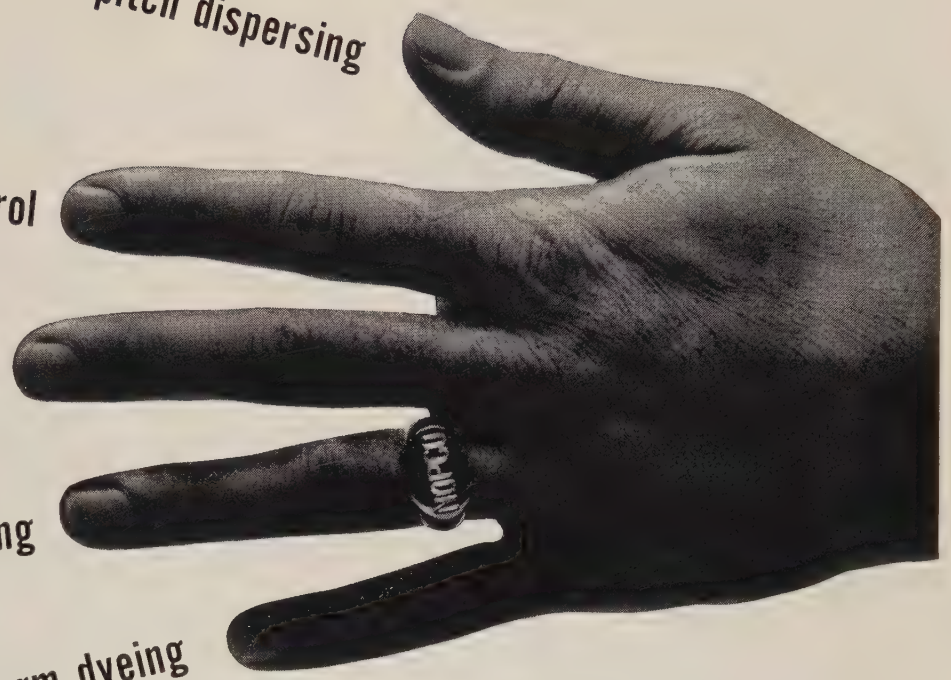

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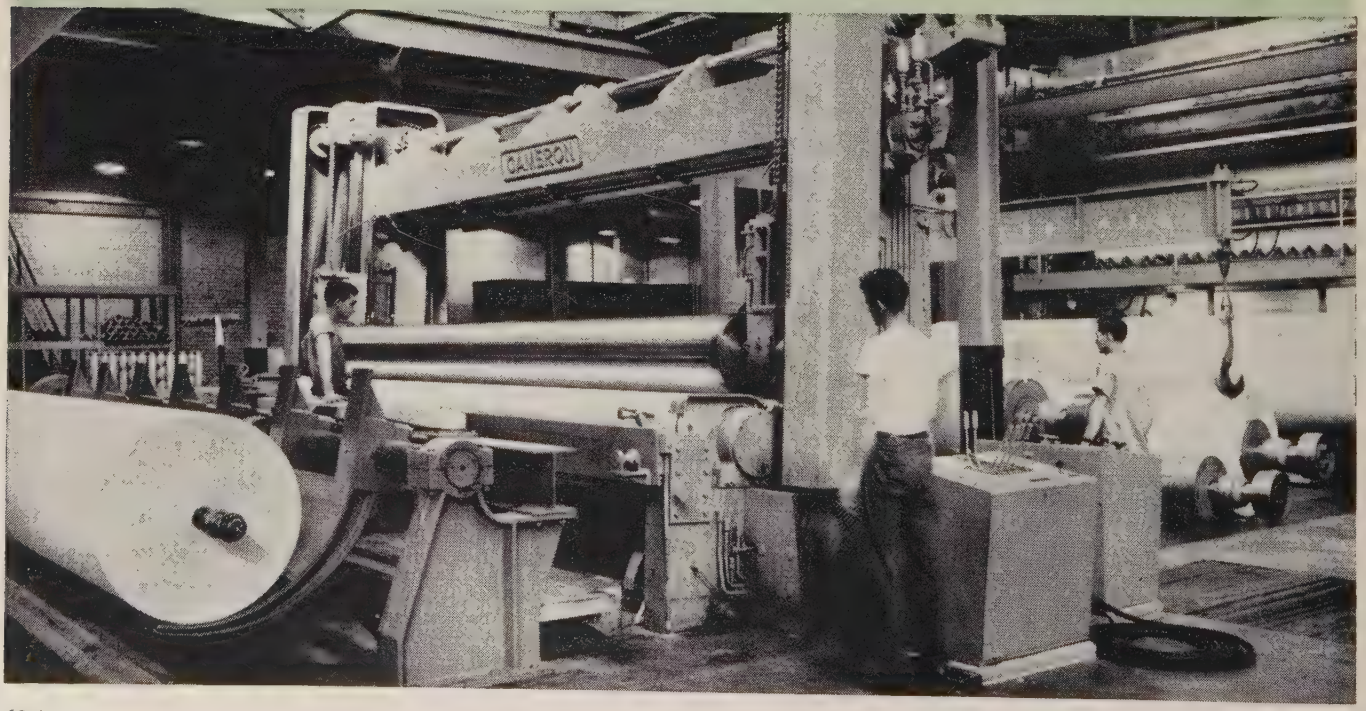
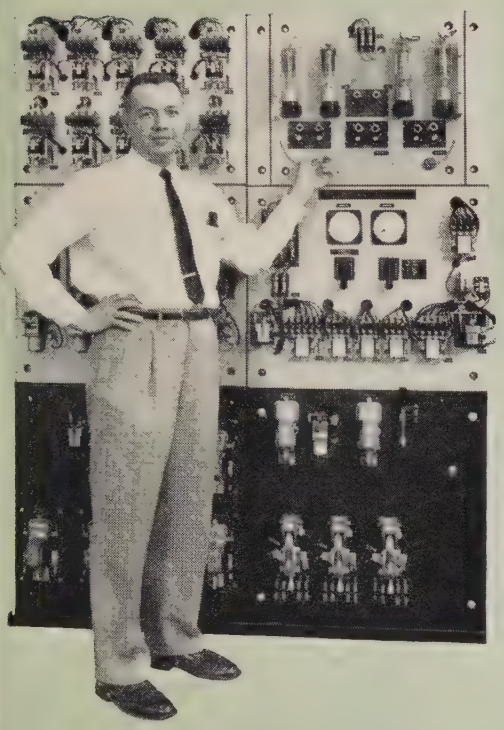


"Reliance drive to power third machine at Marathon Southern



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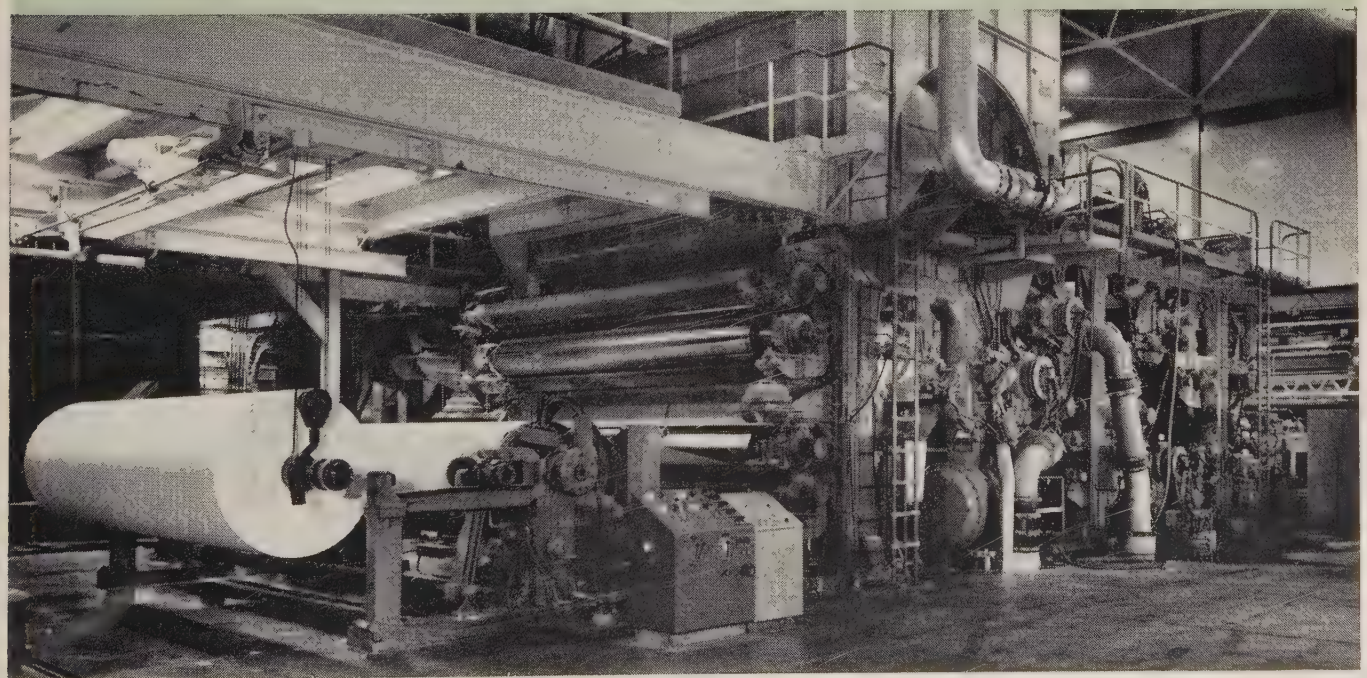

Kesler (*200*) describes how corrosion research and development are handled in the pulp and paper industry. Several suggestions are made for improved efficiency in recognizing and combating corrosion problems within the industry.

Uusitalo (*201*) suggests that alkaline digester corrosion can be minimized or prevented by reducing the sodium hydroxide content of the white liquor in the initial cooking stage or by adding a sufficient amount of polysulfides.

Hopper (*202*) discusses the changes in the cooking operation which have taken place since 1934 and their effect on the increase in rate of corrosive attack on alkaline digesters. Mueller (*203*) reports that anodic protection can be used in stationary alkaline pulping digesters when oxidized black liquor is used for makeup.

Richter and Snyder (*204*) obtained a patent for reducing corrosion which provides for an inner liner spaced from the digester shell and arranged and constructed so that a heat-insulating layer of pulp fibers is accumulated between the liner and the shell. This layer restricts heat flow from the shell so that localized evaporation and concentration of incoming liquor is avoided.

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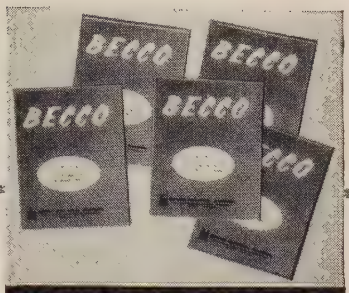

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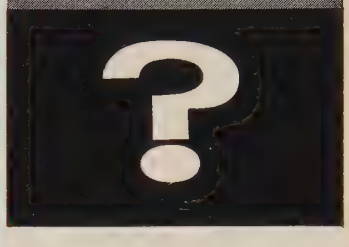

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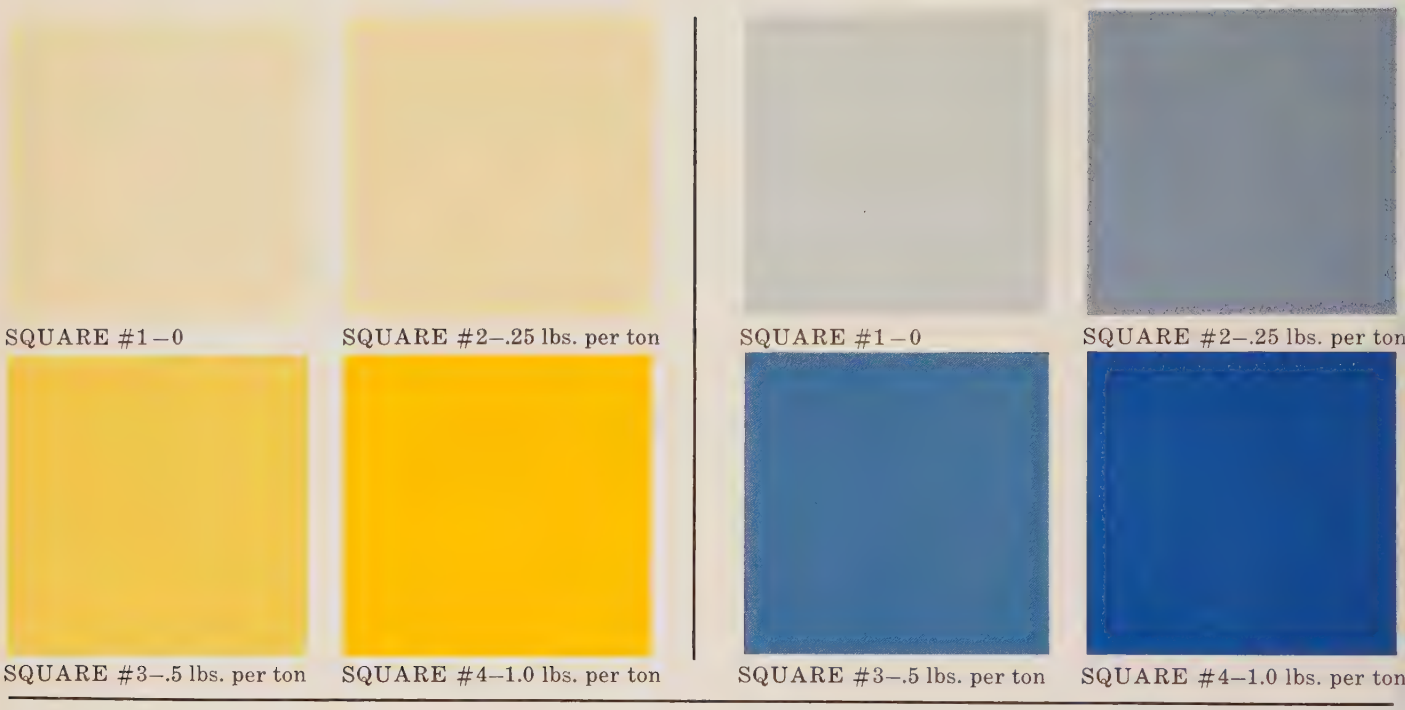
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Latest pilot runs show you can improve coatings using urea formaldehyde concentrate to insolubilize a variety of commercial starches

The addition of U.F. Concentrate-85, Nitrogen Division's urea formaldehyde product, to starch-clay colors formulated with a variety of commonly used starches, produces coated paper with improved wet rub resistance and printability. The paper is comparable in quality to paper coated with butadiene-styrene or acrylic latices.

An added advantage: The preservative action of the contained formaldehyde in U.F. Concentrate-85 retards starch deterioration, sharply reducing unpleasant odors common in hot weather.

These beneficial effects were demonstrated in an extensive series of trial runs on a high-solids pilot plant roll coater operated under plant conditions. The tests were conducted to determine optimum conditions for improved coatings.

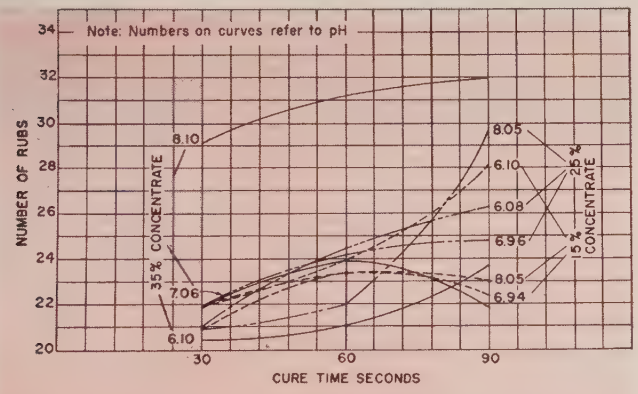

Figure 1 — Effect of cure time on wet rub resistance of starch-clay coatings formulated with Flowkote starch plus varying amounts of U.F. Concentrate-85 and applied at different pH levels.

Variables measured were: pH, curing time, urea formaldehyde concentration and type of starch used.

Starches used were National Starch and Chemical Corporation's Nadex 350, a corn dextrin; Flowkote, an oxidized starch; and Amioca. Adhesive concentration in all cases was 16-18%, based on weight of dry clay.

WET RUB RESISTANCE

Wet rub resistance, determined on American Instrument Co.'s wet rub tester, increases with cure time. How great an increase depends on the starch, the more reactive starch showing the more pronounced effect. Figure 1, on which pH and amount of U.F. Concentrate used in the trials are parameters, shows this relationship for Flowkote.

In all cases, a 25% concentration of U.F. Concentrate-85 (by weight, based on weight of dry starch) gives better results than a 15% addition. However, more than 25% is not generally needed for satisfactory wet rub resistance.

The effect of pH varies, but made no critical difference within the pH range investigated.

PRINTABILITY

Printability, determined by IGT printability tests run on calendered sheets, increases with cure time for a starch such as Flowkote (Figure 2). Figure 2 also shows that for Flowkote, concentration of U.F. Concentrate-85 above 25% decrease printability, and that printability decreases with increasing pH.

With the more reactive Amioca starch, neither cure time nor pH has much effect on printability. However, printability increases with the amount of concentrate added.

ACTION OF U. F. CONCENTRATE

Experimental results show that U.F. Concentrate-85 is composed of methylolureas in equilibrium with free formaldehyde. All the contained formaldehyde is available for further reaction — in this case, to form stable, resinous products involving chemical linkages of the starch and cellulose. These are the same reactions for which coaters use formaldehyde and its methylol derivatives.

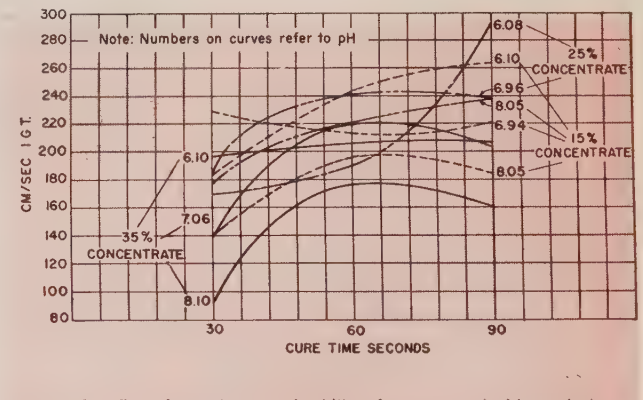

Figure 2 — Effect of cure time on printability of paper coated with starch-clay colors containing Flowkote starch plus varying amounts of U.F. Concentrate-85 and applied at different pH levels.

U.F. Concentrate-85 contains 85% active ingredients — 60% formaldehyde and 25% urea, by weight. You'll find complete information on it in Allied Chemical's 36-page brochure, "U.F. Concentrate-85". Also available is a reprint of an article describing details of its use to improve starch-clay coatings. Write for your copies today. 2819

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46th

Annual Meeting

of the

Technical Association

of the

Pulp and Paper Industry

Commodore Hotel,

New York, N. Y.

Feb. 19–23, 1961

"We offer free mill trials to show the fine results and low cost of groundwood pulp one-stage hydro bleaching—the only cost to the mill is for the hydro"

George V. Rittenhouse (left) and Robert W. Barton

Technical Service Supervisor Bob Barton and District Manager George Rittenhouse, both of Virginia Smelting Company, answer questions about bleaching groundwood pulp with one stage of hydro.

Q Do zinc and sodium hydrosulfites provide the most economical method of one-stage pulp bleaching?

Mr. Barton—If the mill is going for anything between 2 and 11 brightness points, bleaching groundwood pulp with hydro is virtually always the cheapest method. It is easier to apply—there are no control tests involved—and the chemical cost is much lower.

Q Is it difficult to set up a trial run for a mill that might want it?

Mr. Barton—Not usually. Frequently much of the equipment we need is available in the mill. We go in with our lab equipment and make tests, checking the *p*H we're going to deal with, and possibly deciding on the maximum temperature possible. We give all the facts to the mill—and if they want the trial, we set it up. Often the mill has a pulp storage chest. A few changes and we can use it as a bleaching tank. We'll bring in feeders, attach hoses, and start the trial.

Mr. Rittenhouse—Our technical men stay there until the setup is running perfectly and they make sure the operators know everything they need to. You'd be surprised at what we run into sometimes, even in mills that have been bleaching regularly with hydro. They take the powdered hydro and dump it in on top of the stock. Of course, hydro breaks down fast when it is exposed to air, and they wind up with reaction times too short—temperature not showing the proper effect—and poor brightness response at the end. I might add that faulty mixing like this can cause a corrosion problem, too—a problem that does not arise if proper air-free mixing is standard procedure.

Mr. Barton—A good point, George. This is the chief reason why some mills are under the mistaken impression that hydro always causes a corrosion problem. If hydro is handled properly there's no corrosion problem.

Q Isn't a mill trial a pretty extensive service at no charge?

Mr. Rittenhouse—Yes, I suppose so—but I consider it the most effective way of showing a mill the economy and the good results obtainable with hydro. After all, mill trial data offer the most believable evidence of what I say. And production doesn't have to be stopped—we can almost always set up the mill trial without affecting it.

Q What are the advantages to the mill in using a "Virginia" hydro?

Mr. Barton—There are several. One is the consistently uniform quality and purity of our hydro, either the zinc or sodium. It's a very clean, free-flowing powder—made, packed and shipped as moisture-free as modern manufacturing methods can make it.

Mr. Rittenhouse—And of course our technical service goes with it. A mill can always call for assistance and get a "Virginia" technical man. It's the accepted thing with our customers.

• • •

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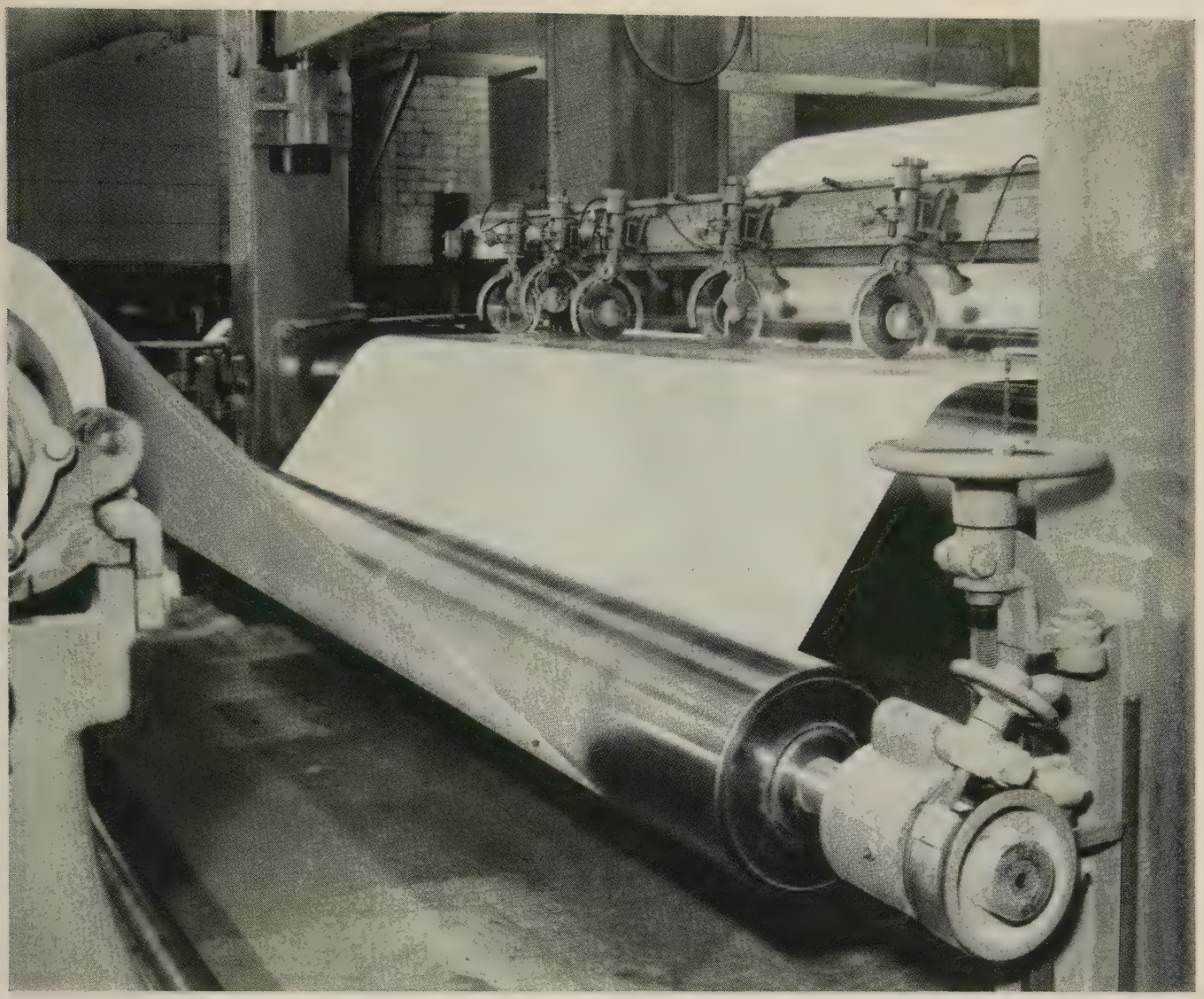

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Improved slitter arrangement

The slitter arrangement pictured above materially increases slitter life and makes slitter settings simpler and faster. This new slitter design incorporates a preset shear angle between top and bottom slitter, plus other improvements which result in a 200 to 300 per cent increase in slitter life. This new arrangement also insures correct slitter setting, with a 50 per cent reduction in slitter resetting time and less attention from the operator. This arrangement is the result of two years' development and is now standard on all Beloit winders.

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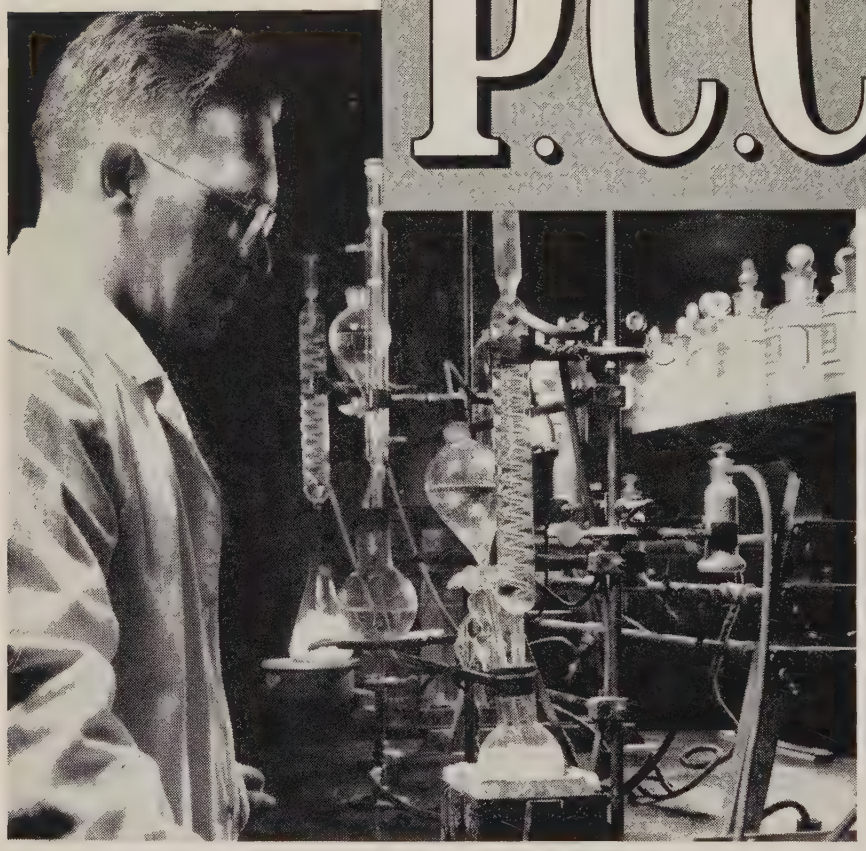

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A Nalco PRODUCT MANAGER

Answers Basic Questions on Coagulation of Solids in Potable, Process and Waste Waters

New Chemicals and New Techniques Mark Advances in Solving A Frequent Plant Operating Problem

Improved methods of chemical clarification are constantly being developed, tested, and put into full-scale commercial use. Here are answers to some of the questions we hear frequently in this area of Nalco service activity.

Question: What is coagulation?

Answer: Coagulation is the agglomeration—chemical gathering together—of the very fine solid particles suspended in water. These particles create turbidity, coloring, and some types of odors.

Question: What are the advantages of coagulation over sedimentation?

Answer: Speed, more effective clarification, and smaller capital equipment investment are all advantages of coagulation over sedimentation.

Question: Are all coagulation requirements the same?

Answer: No. In some cases, a high degree of sparkling clarification is essential. In others, a lesser degree of clarification is satisfactory. Sometimes, the removal of color is the only job to be done Seasonal and other changes in water and turbidity characteristics also change the type and amount of treating necessary. Overall quality of the water must be compatible with the end-use requirements—not based on a fixed "cure-all" treatment program.

Question: What are the characteristics of a good coagulation program?

Answer: Continuous production of water of the necessary clarity and quality to meet end-use requirements; with the flexibility to handle changing conditions in input water characteristics. A good coagulation program should also increase the length of filter runs.

Question: Historically, what coagulant chemicals have been used?

Answer: Most common chemicals used as coagulants were: iron salts, alum (aluminum sulfate), and lime (calcium hydroxide).

Question: What are some of the more recent improvements in the field of coagulation?

Answer: Sodium aluminate is probably the first of the "modern" coagulants. It supplies an alkaline source of alumina with a negative (anionic) charge, and constitutes a coagulant which ideally meets the requirements of principal coagulation process uses—frequently with alum, to improve flocculating and give faster settling rates at lower overall costs.

Natural and synthetic organic polymers are the most recently-developed coagulant chemicals. They are high molecular weight materials which are anionic, cationic, or nonionic; and function as high-efficiency coagulant aids. Or, as is true with the cationic polyelectrolyte developed by Nalco, they may function as both coagulants and coagulant aids. Use of these materials provides greatly increased floc particle size and weight, resulting in increased clarity, longer filter runs, and increased throughput rates.

Question: Will any of these newer coagulant chemicals solve all clarification problems?

Answer: No. There is no all-purpose coagulant chemical; nor is there likely to be one. Too many variables enter into the choice of proper chemicals to fit specific operating conditions: from the type of influent water through kinds and arrangements of equipment to the clarity needs of effluent water.

J. T. Burke, Manager
Nalco Coagulation Chemicals Department

Question: What is the best approach to solving a specific coagulation problem?

Answer: Take all the factors involved, and accumulate specific information on them: (a) Make a thorough investigation of the supply water, the equipment employed, the end use of the water, and the performance characteristics of chemicals available for coagulation and coagulation aid. (b) Test coagulation with various chemicals by beaker tests and other laboratory evaluation techniques. Evaluation should include analysis of waters produced, to make certain that the quality is compatible with end-use requirements. (c) Get the advice and assistance of experts as to methods and locations of applying chemical treatments to get maximum coagulation efficiency.

Question: Does Nalco provide the experts to gather this information?

Answer: Yes—in addition Nalco supplies the most complete line of coagulants and coagulant aids in the industry.

Question: How is Nalco qualified to help solve coagulation problems?

Answer: Nalco's basic background is successful water treatment. The Coagulation Chemicals Department at Nalco is made up of "specialists' specialists": men whose sole job is to provide Nalco Representatives and their customers with the materials and assistance necessary to solve coagulation problems.

No other organization has more experience or better facilities to solve *your* coagulation problem, promptly and economically. Call or write for action now.

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Via Conveyors

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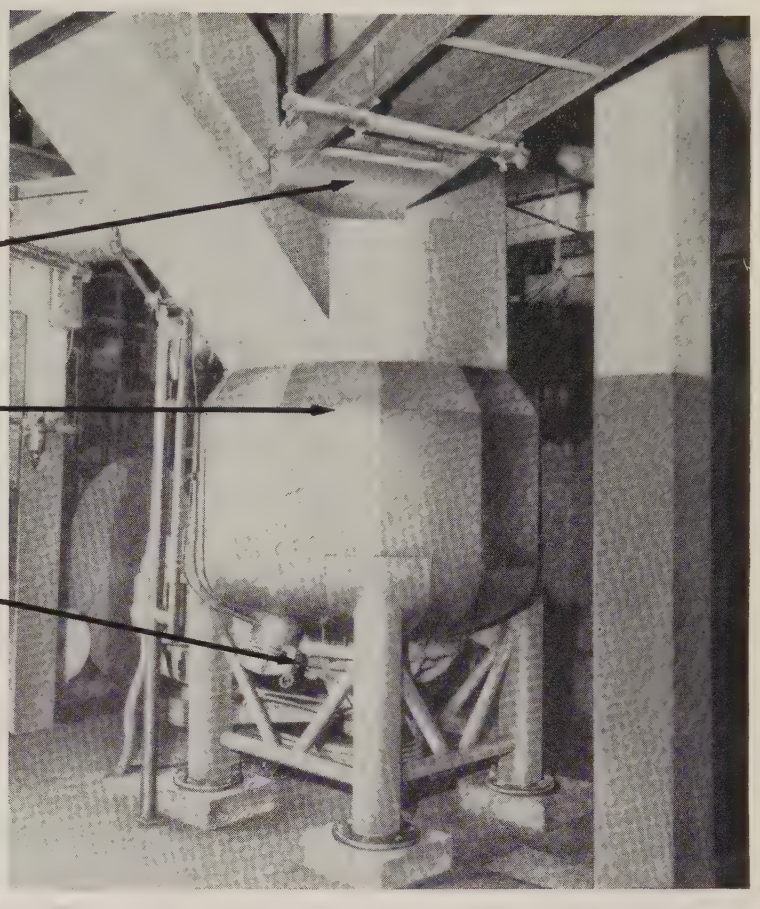

With the Impco Solvo® Pulper

The Model 2U Solvo Pulper, playing a dual roll at Bird & Son, Inc., East Walpole, Massachusetts, has sharply reduced previous material handling costs. It is operating continuously under automatic control as both a material handler and a pulper. Boxboard broke from the converting machines is delivered by conveyors to the Solvo which simultaneously pulps and pumps it to the pulp mill. The units' single 50 HP motor handles both pulping and pump-away demand at a 24 ton/day rate.

This is one of a wide variety of Solvo Pulper applications. Others include de-ink cooking, dry end broke handling, paper machine furnish pulping, transfer of high yield cooked chips to refiners as well as the normal pulping of broke, wet strength and waste paper. Units are available for any capacity requirement.

*The pulping element, extractor plate and stock pump are one rotating assembly.

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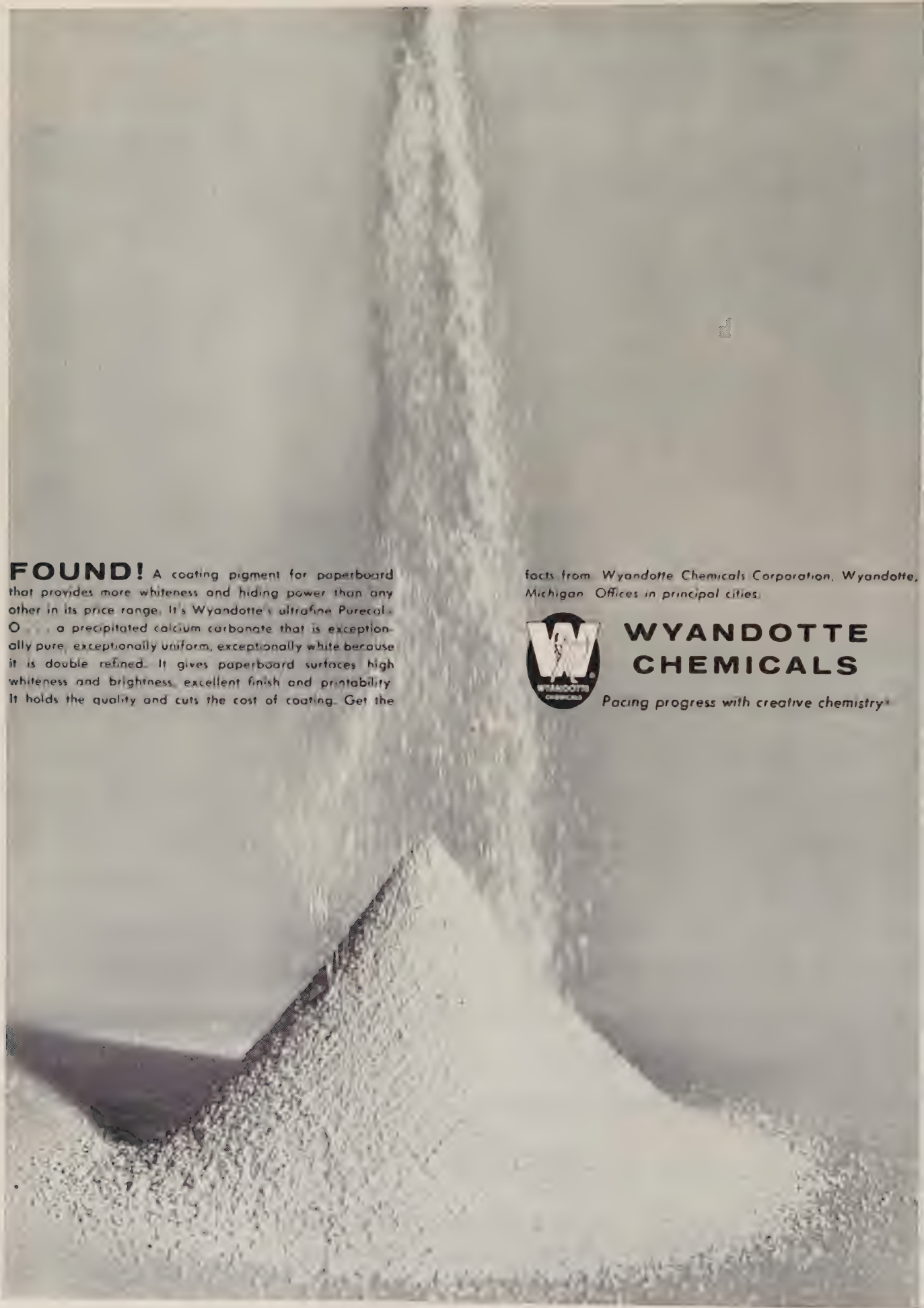
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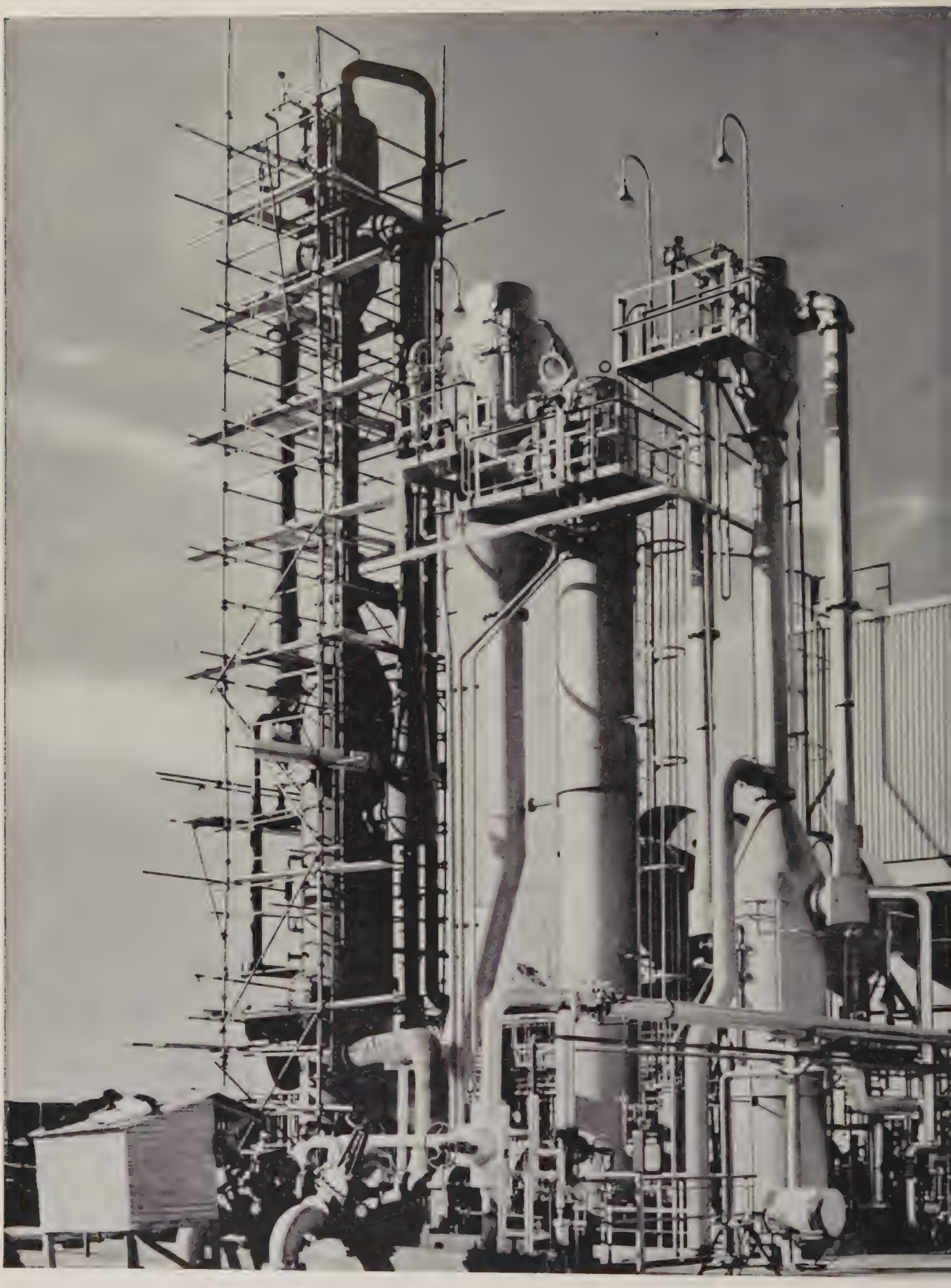

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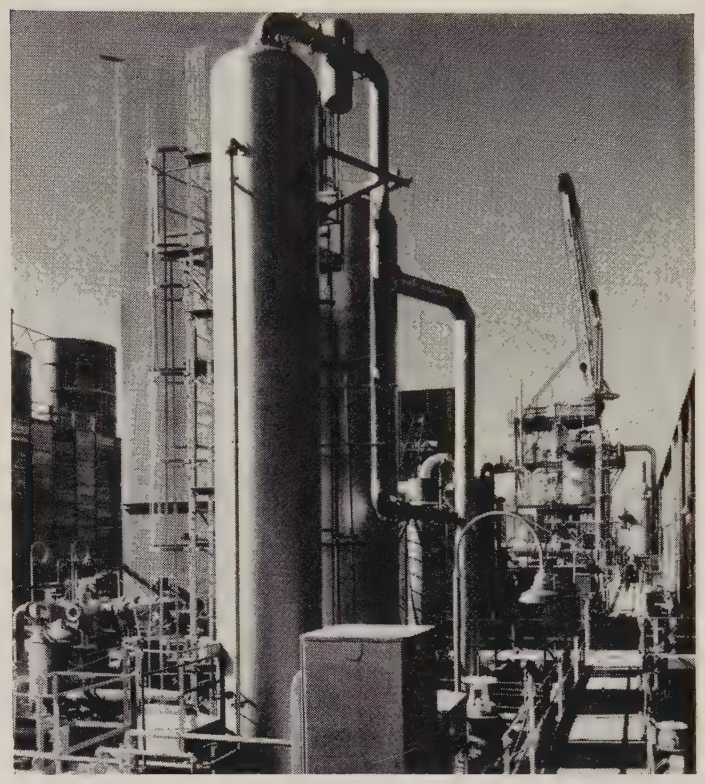
Two other important paper chemicals—sodium peroxide and ammonia—are also available from U.S.I. Sodium peroxide is produced at Ashtabula, Ohio; ammonia at Tuscola, Illinois.

With U.S.I.'s latest polyethylene resin plant expansion at Houston, Texas—and facilities at Tuscola, Illinois—the company's total polyethylene capacity will soon reach 300 million pounds per year. This will make U.S.I. the world's second largest producer.

As part of the company's program to assist polyethylene customers, U.S.I. has come up with significant developments in resins for the paper industry. For example, PETROTHENE® 205-15—one of four new U.S.I. resins for paper coating—exhibits greater greaseproofness at any given coating weight than other resins of similar melt index and density. All four resins are tailored for high adhesion, and reduced neck-in, smoking and polymer build-up at the die.

U.S.I. has also pioneered the new technique for producing crystal-clear cast polyethylene film, and has just released new resins for this purpose. More and more converters are packaging with polyethylene now that the clarity problem has been solved, because of the enhanced shelf life which this film gives to paper products.

U.S.I. has a pulp-to-package interest in the paper industry's growth. To take advantage of our research and development work, large capacity, prompt technical service, write or call your nearest U.S.I. sales office, or company headquarters in New York.



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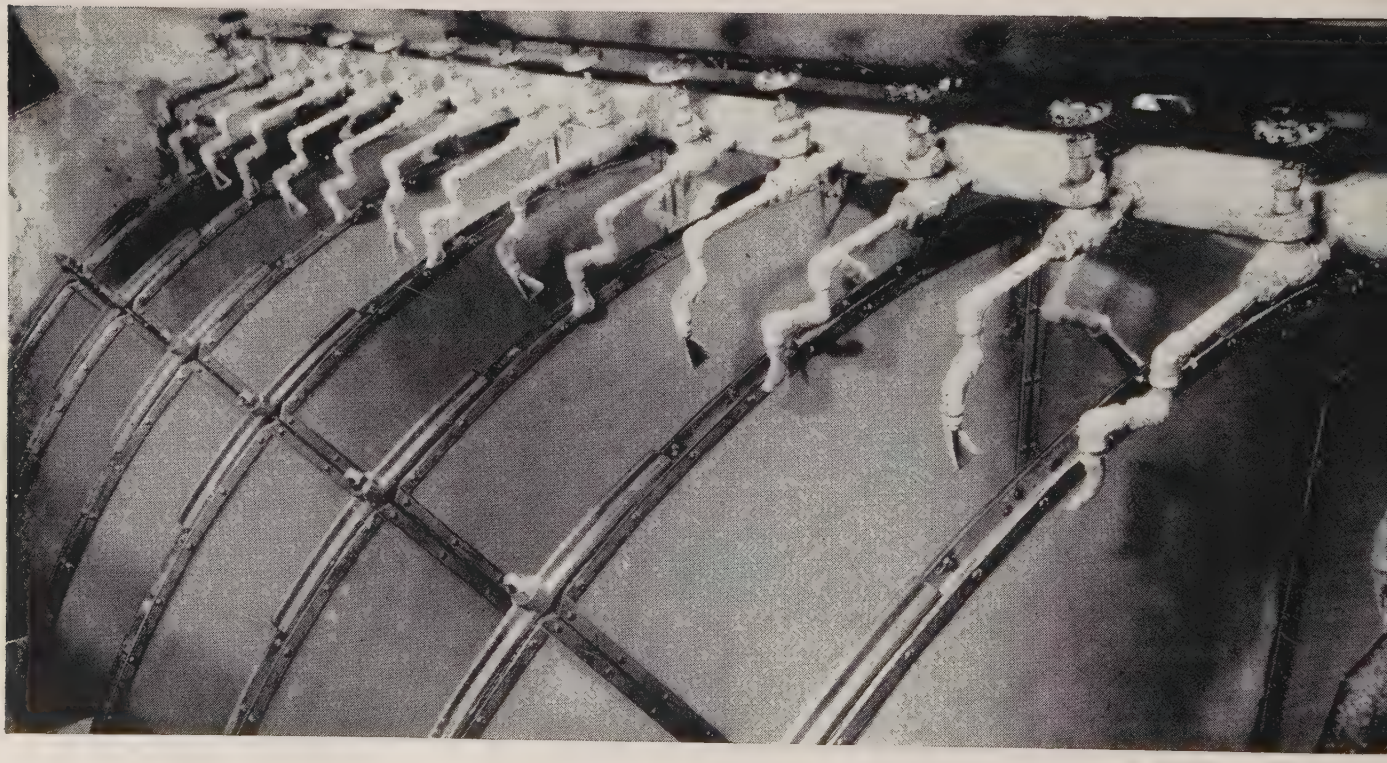
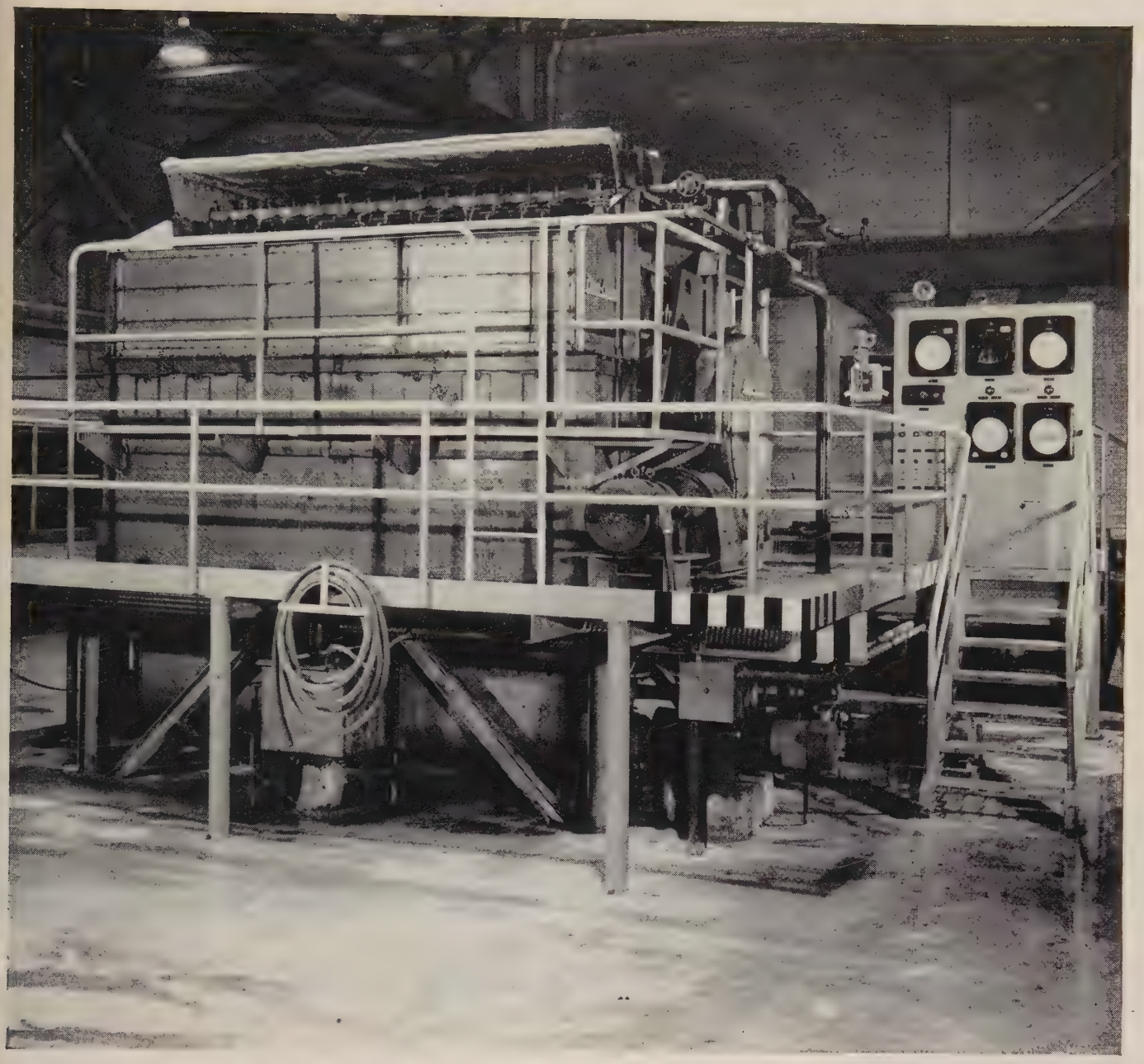
Another example of the paper industry's wide acceptance of the American disc-type Saveall for maximum recovery of fiber from white water is seen in this installation at the well-known Hammermill Paper Company's Erie, Pennsylvania, Division.

The equipment handles white water from Hammermill's No. 7 machine, producing various grades of papers including Hammermill Bond and Hammermill Offset. Maximum flow is 1,200 g.p.m. Effluent ranges from 0.0 to 0.25 lb. per 1,000 gallons, showing the high rate of recovery achieved.

As well as excellent performance, the American Saveall offers the advantage of small space requirements, easy and quick replacement of filter disc sectors and fully automatic, continuous operation. Two sizes are available—9 ft. dia., with a maximum of 16 discs having 1,790 sq. ft. filter area and occupying 28 ft. x 12 ft. floor space; and 7 ft. dia. with a maximum of 10 discs and 700 sq. ft. filter area, requiring 9 ft. x 17 ft. floor space. For more information, write for Bulletin 3306 to Dorr-Oliver Incorporated, Stamford, Connecticut.





▲ Installation at Hammermill illustrates compact design and small floor space requirements of American Saveall.

◀ Close-up shows easily removable disc sectors and new "less splash" sluicing nozzles for continuous removal of recovered fibers, which are then returned to the stock chest.

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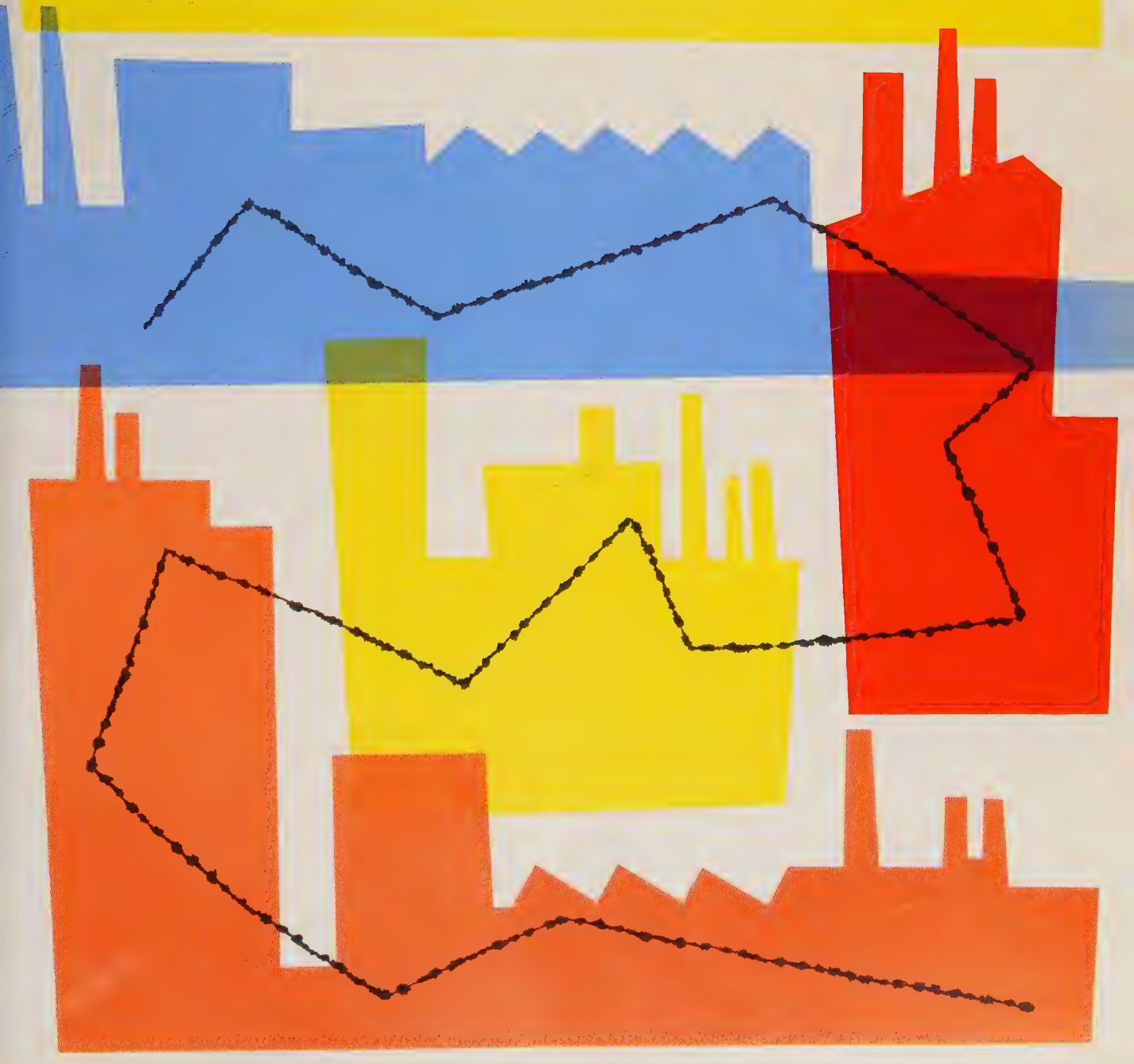
LET YOUR APPLETON MAN TAKE A FRESH LOOK AT YOUR FELT APPLICATION AND PERFORMANCE PROBLEMS. It can be a rewarding experience—it has been for others. By working with our laboratory research men, felt designers and experienced production people, he can bring you a custom designed application to improve felt performance and life. What better way to increase production and cut costs (has a nice effect on profits, too). Ask him to take a look-see soon! You'll be glad you did!

AP-PLY FELTS
Appleton Woolen Mills
Working With The Paper Industry
Appleton, Wisconsin



Solophenyl™
Blue 8 GLP

A most fitting background for type

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Dyestuffs by Geigy.

Here's a new approach to Blue and Green bond and printing papers.
Solophenyl Blue 8 GLP
A low cost direct blue with the necessary fastness properties for fine papers.

Paper Bond Blue BP
A low cost Alizarine type blue with rapid developing characteristics and needed properties for high grade printing papers.

Use the above two blues as a base for blue shades or in conjunction with
Chinoline Yellow P Conc
Diphenyl Fast Yellow 4 GP
for green shades.
Our technical staff will be glad to demonstrate the adaptability of those products.

Geigy
Division of Geigy Chemical Corporation
Saw Mill River Road
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WONDERWHITE:

FOR THE BEST IN BRIGHTNESS AND FORMATION

GBS+ClO$_2$ +R&D=

WONDERWHITE

You probably have heard paper manufacturers commenting on how WONDERWHITE achieves "high brightness without loss of opacity." But did you know that you also get this brightness in a fiber with unexcelled paper-making qualities?

Paper makers have always known that Northeastern Spruce has unexcelled paper making properties. Now, with WONDERWHITE, you have the forming and printing qualities available only in a spruce sulphite, plus the high brightness which only Chlorine Dioxide can give.

Maybe this pulp is the answer to a number of your problems. May we suggest a trial shipment? You'll soon see why we called it WONDERWHITE.

GASPESIA SULPHITE COMPANY LTD.
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SOLD BY: **Anglo Paper Products, Ltd.**
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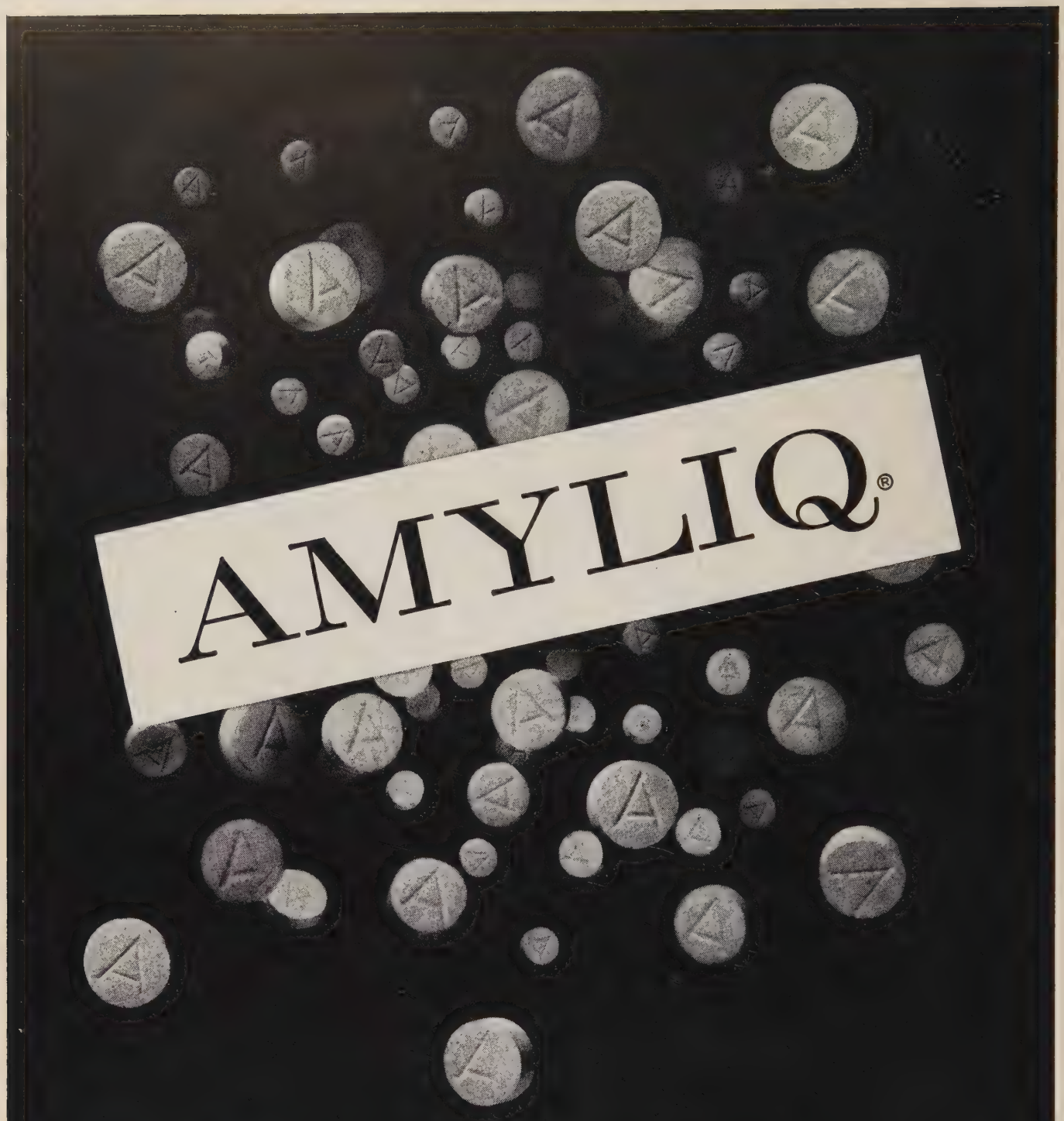

For Starch Conversion...

The α-amylase for the paper industry...available as AMYLIQ Super and AMYLIQ Concentrated bulk powders–and as AMYLIQ tablets, readily soluble preweighed units costing no more than powder.

For broke recovery: SERIZYME® liquid proteolytic enzyme

WALLERSTEIN COMPANY Division of Baxter Laboratories, Inc.
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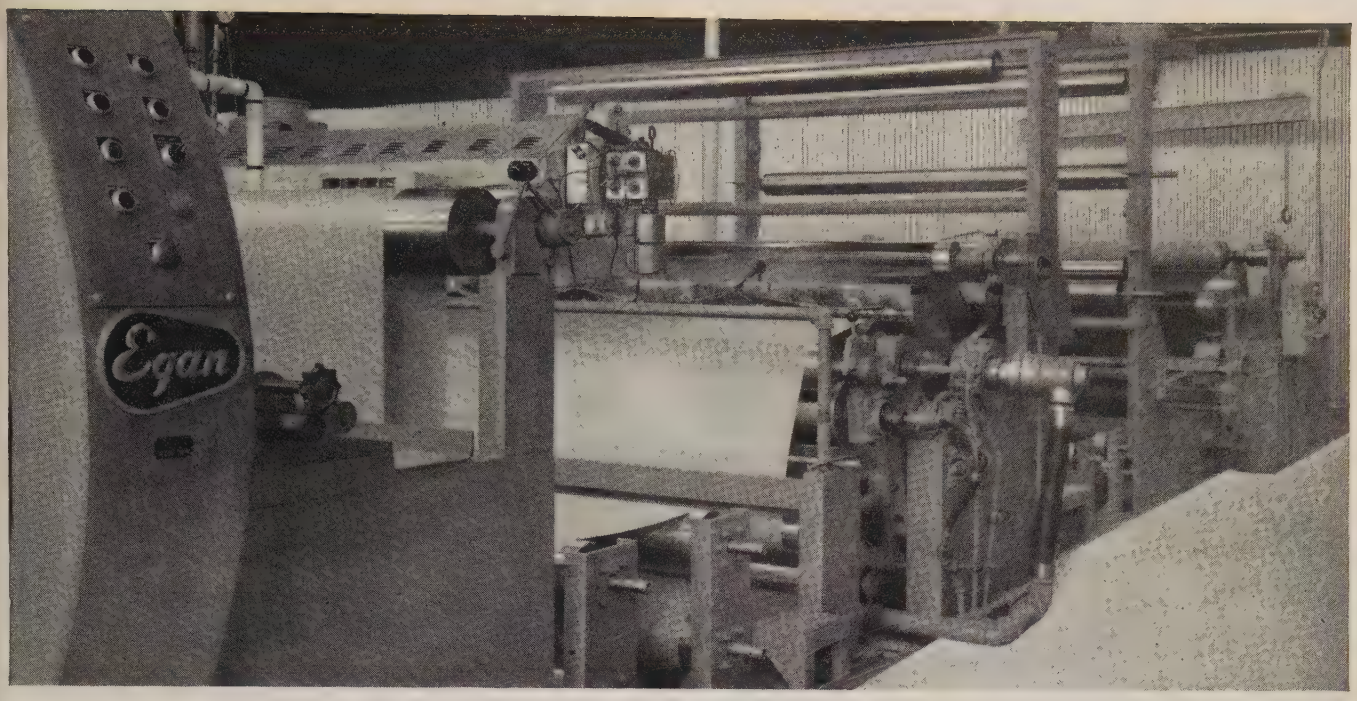


FOR HIGH-SPEED POLYETHYLENE COATING OF WIDE PAPER

CONTINUOUS OPERATION AT 1,000 FPM

When the converting operation calls for polyethylene coating of wide-web kraft and other papers, no production unit can surpass an Egan Extrusion Coater!

The coater illustrated above includes a flying-splice unwind stand, direct electric heat extruder, die, laminator, and single drum surface winder. The combination is capable of continuous operation at 1,000 fpm.

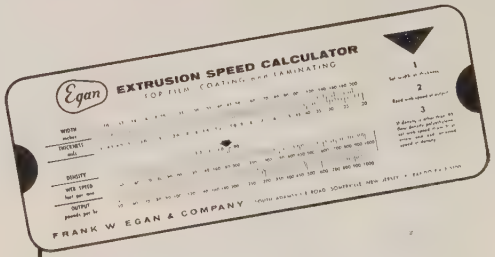
Open construction of the laminating unit permits maximum access and visibility for the operators. The laminator also has controlled stripping tension, and includes *patented* horizontal and vertical adjustments of the laminating nip which are essential for maximum adhesion control.

The cooling roll is of a unique *patented* design. This double spiral roll provides for highly efficient cooling and extremely uniform temperatures across the roll. Use of the double spiral permits water to enter simultaneously from both ends of the roll and exit at opposite ends from the point of entry. The temperature across the entire face of the roll during operation has a differential of no more than 2°F.

Egan production units are available in widths to 120″.

WORLD'S LEADING MANUFACTURER OF EXTRUSION COATERS

WRITE for this handy Egan Extrusion Speed Calculator which quickly determines any extrusion capacity required for a specific web speed, width, and coating thickness.



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Egan FRANK W. EGAN & COMPANY
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MANUFACTURERS OF PROCESSING MACHINERY FOR PAPER, FILM & FOIL, AIR DRYING SYSTEMS; ROTOGRAVURE PRINTING PRESSES; PLASTICS EXTRUDERS & ACCESSORIES; TEXTILE FINISHING MACHINERY.

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OVER 80 YEARS' EXPERIENCE

in pulp and related services

Information cannot be inventoried—it is too hard to keep and too perishable. Yesterday's data may be as obsolete as last year's. Still, information on supplies, on prices, and on production is management's most valued tool. For information on pulp and pulp-type products, telephone C & O. One call checks many sources. Call "C & O".

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Representing:

Champion Paper & Fibre Co.

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A/S Hurum Fabriker, *Bleached Sulphate*

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A/S Krogstad Cellulosefabrik, *Bleached Sulphite*

A/S Tofte Cellulosefabrik, *Bleached Sulphite*

A/S Toten Cellulosefabrik, *Bleached Sulphite*

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Agents for:

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Distributors for:

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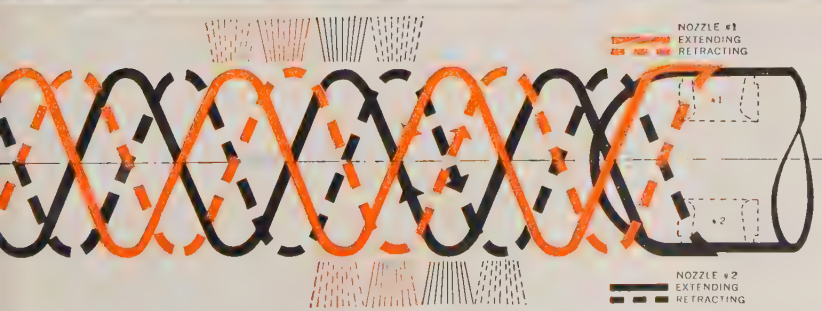
Diamond developed for more economical power...

A CLEAN SWEEP...EVERY INCH OF THE WAY

Get uniform, complete cleaning in those "tough-to-clean" zones. Specify Diamond's IK-300 Retractable Blower. The exclusive close-spaced IK helix assures a penetrating nozzle sweep every inch of travel regardless of tube bank arrangement and tube spacing. What's more, because sectionalized lance tube construction cuts deflection to a minimum, smaller boiler cavities are possible. In addition, a unique method of equalizing nozzle end-thrust assures "minimum-wobble" travel regardless of distance.

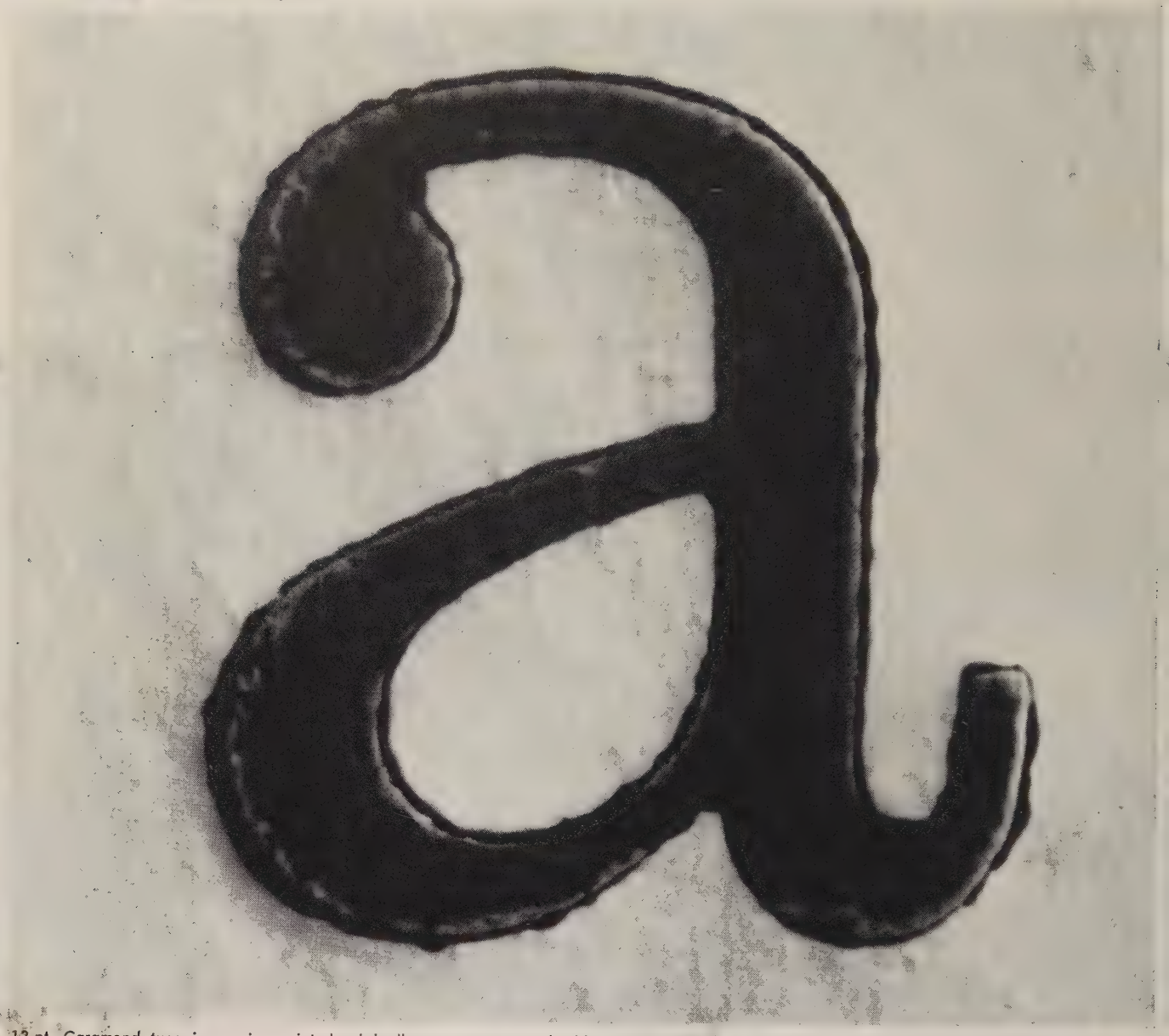
IK's are just part of the reason behind Diamond's established superiority in boiler cleaning systems. Add a complete line of cleaning equipment, an experienced, imaginative engineering staff and an unmatched service organization. No wonder thousands of utility and industrial power plants are equipped with Diamond Cleaning Systems — engineered and designed to provide you with more economical power.



Diamond

DIAMOND POWER SPECIALTY CORPORATION, Lancaster, Ohio • Diamond Specialty Limited, Windsor, Ontario

DOW | LATEX



12-pt. Garamond type impression printed originally on paper coated with Dow Latex 630 and magnified approximately 85 times to show paper surface uniformity. The uniform impression and ink distribution greatly improve print quality.

Now . . . better control of paper uniformity with **NEW DOW LATEX 630**

- **Excellent Mechanical Stability!**
- **Superior Pick Resistance!**
- **Excellent Starch Compatibility!**

Three good reasons why new DOW LATEX 630 can help you make . . . help you sell . . . better paper than ever! You'll find it far easier to produce uniform paper with smoother, more pick resistant coatings made from Dow Latex 630. Paper surfaces provide a new high in printability, reproducing both illustrations and type with remarkable sharpness and realism. Dow Latex 630 will give you the coating quality you usually associate with higher cost!

Contact Dow now for detailed information to help you make the most of new DOW LATEX 630! Write to THE DOW CHEMICAL COMPANY, Midland, Michigan, Coatings Sales Department 1934FC10.

See "The Dow Hour of Great Mysteries" on TV.

THE DOW CHEMICAL COMPANY · MIDLAND, MICHIGAN

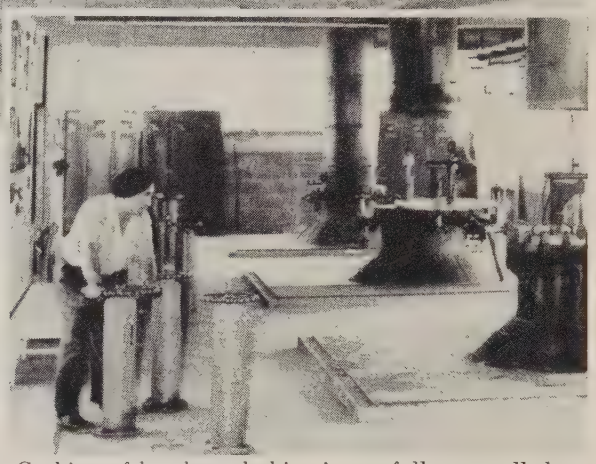
The Cellulose d'Aquitaine mill in Southern France consumes as its principal raw material heretofore unused hardwood species of the area, producing from them high quality bleached pulps.

First pre-hydrolysis kraft pulp mill in France

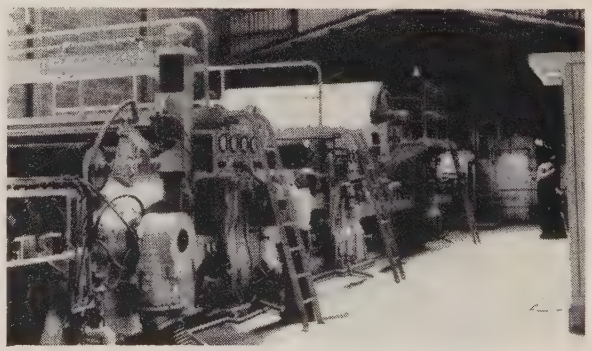
Making France self-sustaining in the production of nitrating grades of cellulose, the mill of Cellulose d'Aquitaine, St. Gaudens, has been in operation since the summer of 1959 and produces up to 120 tons of nitrating pulp or 170 tons of bleached paper grade pulp per day.

The Parsons & Whittemore-Lyddon Organization had full responsibility for design and construction of the plant. It is but one of fourteen mills now in various stages of development for which the Organization provides technical supervision, complete equipment and other important services under a global contract.

Parsons & Whittemore-Lyddon's staff of renowned pulp and paper making experts is backed by the Organization's international machinery manufacturing and sales facilities. Our experts are prepared to assist you in planning, designing, financing, constructing, equipping and operating pulp and paper mills in any country having suitable wood or agricultural fibers. For full information on any or all of these services, write to our nearest office.



Cooking of beechwood chips is carefully controlled on this digester operating floor. Three of the four digesters are shown. All of them are constructed with special alloy lining to permit a pre-hydrolysis cooking stage at extremely low pH values.



The 136" wide Fourdrinier pulp drying machine, built by Black-Clawson, includes cantilever design, modern vacuum equipment and hydraulic press controls.

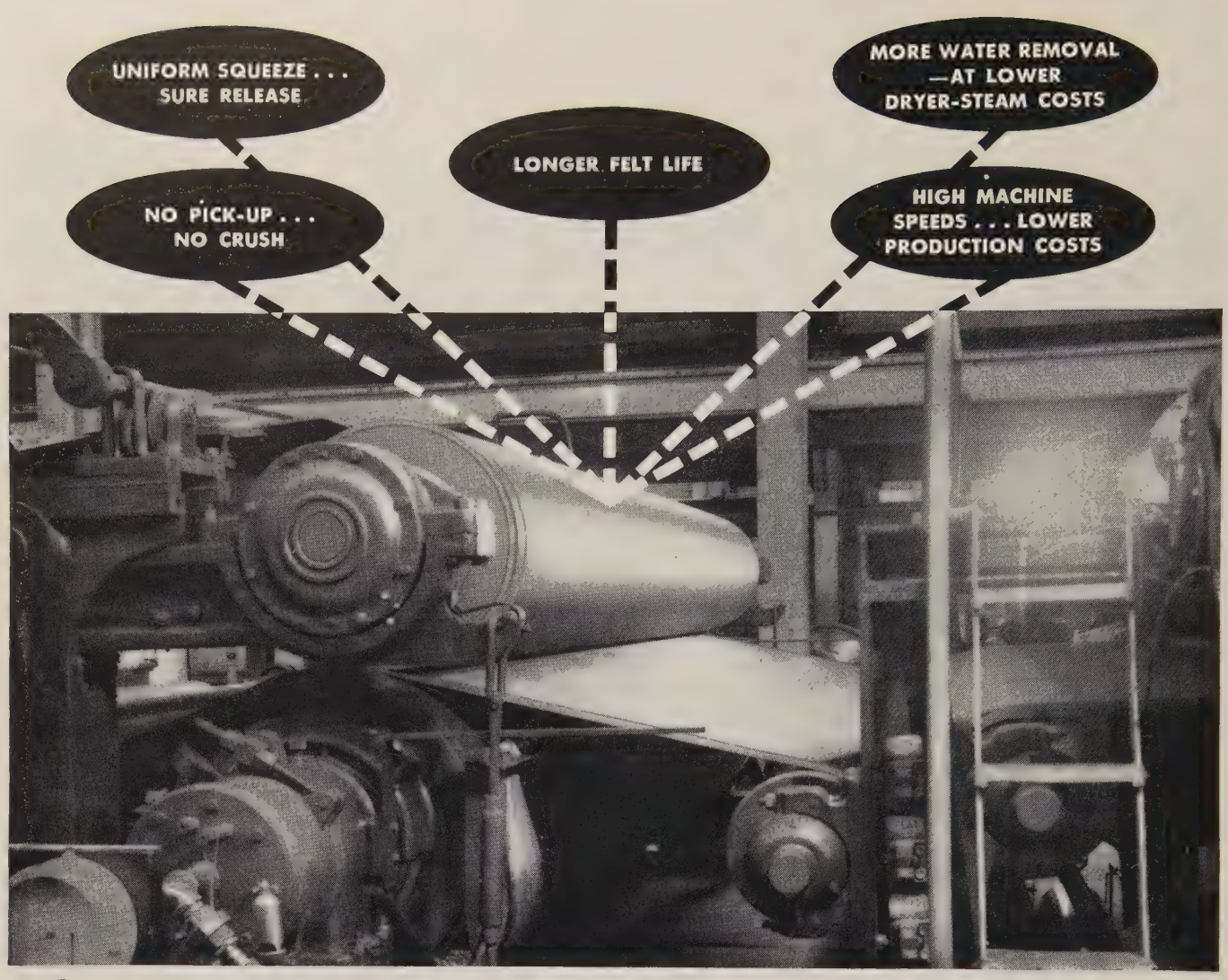
THE PARSONS & WHITTEMORE / LYDDON ORGANIZATION

World leaders in the development of pulp and paper mills for the use of local fibers

18-19 Savile Row, London W. 1, England

5 Rue Jean Mermoz, Paris 8°, France

250 Park Avenue, New York 17, N. Y.


UNIFORM SQUEEZE . . . SURE RELEASE
MORE WATER REMOVAL —AT LOWER DRYER-STEAM COSTS
LONGER FELT LIFE
NO PICK-UP . . . NO CRUSH
HIGH MACHINE SPEEDS . . . LOWER PRODUCTION COSTS

Get all these Performance Advantages

WITH MANHATTAN RUBBER COVERED ROLLS!

Advanced roll covering facilities and skilled craftsmanship at Manhattan assure trouble-free roll performance—at lower costs—for every stage of your papermaking operations.

- **An inseparable bond of rubber-to-metal eliminates cover separation**
- **Cover compounds won't crack, corrugate or oxidize**
- **Advanced grinding and vulcanizing facilities insure uniform, exact density—accuracy of crown dimensions within .002''**
- **Rolls require minimum regrinding—even under extreme conditions of use**

Manhattan was first to dynamically balance rolls electronically at customers' machine speeds . . . to assure *even* pressure and moisture distribution in the stock, less wear on felts, bearings and journals. Let a Manhattan roll specialist show you how to get "More Use per Dollar" with Manhattan Rubber Covered Rolls.

SPECIFY MANHATTAN RUBBER COVERED ROLLS FOR EVERY CRITICAL FUNCTION

• Press Rolls—Ray-Roc, Rub-Roc, Sham-Roc, Self-Skinner • Suction Press and Coating Rolls
• Sealed-End Couch Rolls • Marblex Table Rolls

RM1050

ENGINEERED RUBBER PRODUCTS ... MORE USE PER DOLLAR

ROLL COVERING PLANTS AT PASSAIC, N. J. • NEENAH, WISC. • N. CHARLESTON, S. C.

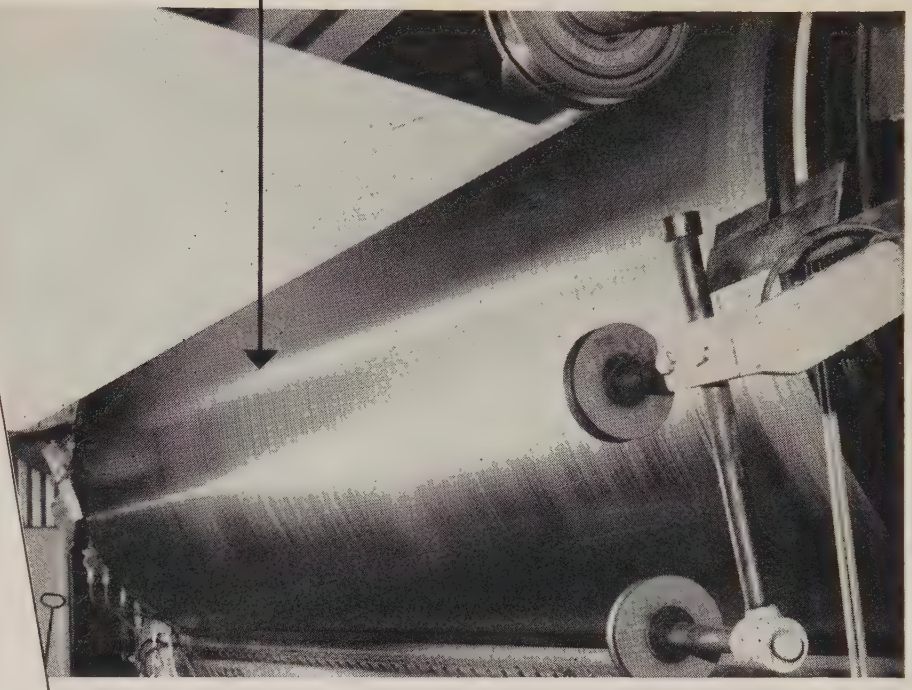
RAYBESTOS-MANHATTAN, INC.

MANHATTAN RUBBER DIVISION, PASSAIC, NEW JERSEY

FURTHER PROOF OF PERFORMANCE

MANDO installs second CRODON-plated sweat dryer

CRODON
The Chrome Plate
TRADE MARK REG U S PAT OFF



"After watching our #4 chromium-plated dryer for about a year and a half, and also watching #2 dryer, I am happy to tell you that these dryers are doing us an awful lot of good.

Whenever we sweated these dryers, we were continuously in trouble with rust streaks in our sheet. This has completely disappeared."

MINNESOTA AND ONTARIO PAPER COMPANY

. . . this frank expression of satisfaction by Minnesota and Ontario Paper Company is impressive evidence of CRODON performance.

In related applications, at least 43 mills are operating more than 100 CRODON-plated dryers in ALL positions, from baby-dryer size to 12′ diameter. One mill has CRODON-plated all dryers in two sections on one machine. Another mill is still operating a CRODON dryer after-size press following 29 years of successful service.

Reduced production loss, improved finish, virtually no maintenance expense — that's what CRODON-plating has meant to dryer users. Let us tell you more about it. Write for details or request a visit by our field representative.

CHROMIUM CORPORATION of AMERICA

Executive offices: 100 Park Ave., New York 17, N.Y.

Plating plants for paper mill equipment:
346 Huntingdon Avenue, Waterbury 20, Conn. 4645 West Chicago Avenue, Chicago 51, Illinois

The KADY® MILL

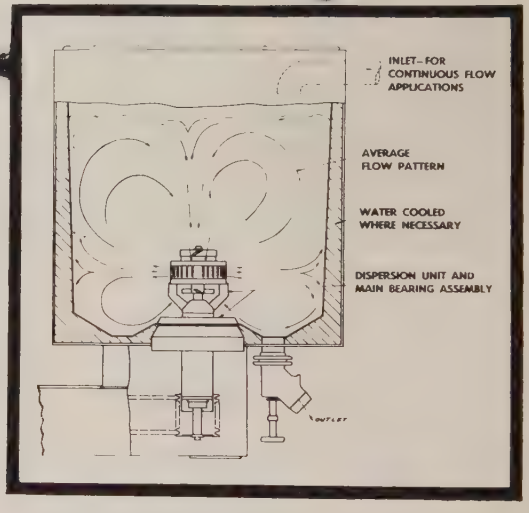
COATING COLOR

in

MINUTES

instead of

HOURS



- ★ **Cook Protein, Casein or Starch in presence of pigment efficiently and economically.**
- ★ **Color for Machine or Off Machine coating in one operation.**
- ★ **Low Viscosity with high solids.**
- ★ **Reduce Binder percentage.**
- ★ **Develops optimum opacity and gloss.**

Over 50 paper plants have approved the advantages and economy of coating color production by the KADY method. The uniformity of viscosity, optimum value from Ti 0_2, calcium carbonate, satin white and from whichever adhesive or combination of adhesives used, prove the superiority of the KADY method to any other.

If you want continuous processing or have a color system deaerating problem to overcome, let us give you the complete story of KADY applications to suit your specific requirements. Many quality control problems on all types of coaters are being solved effectively, color application improved and superior finish obtained by a simple, low cost installation of a KADY MILL!

** Pictured above is the 750 gallon model 600B. Other models, including the new T series, in capacities from ½ gallon on.*

Convenient Lease or Time Payment Plan

KADY® proven best in WORLD WIDE installations:

- United States
- Canada
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Kinetic Dispersion corp.

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a wealth
of wet strength
with

SCRIPTITE* 40

A cationic urea-formaldehyde resin, Scriptite 40 imparts a high degree of wet strength to paper, with less resin add-on. In addition to raising tensile and wet bursting strength, Scriptite 40 also improves dry tensile strength, dry mullen, wet rub resistance, internal sizing, pick resistance, dry stiffness, dimensional stability, and folding resistance. For technical assistance, samples, and literature, write to Monsanto Chemical Company, Plastics Division, Room 779, Springfield 2, Massachusetts.

Monsanto

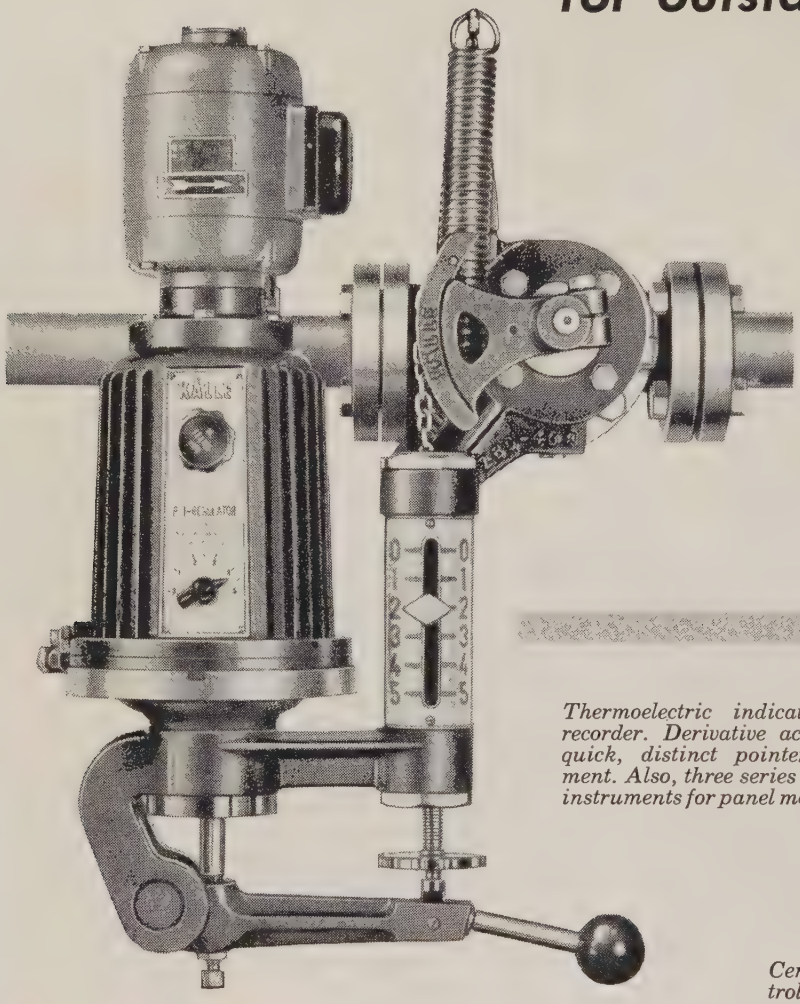
MONSANTO DEVELOPER IN **PLASTICS**

The Monsanto line of paper resins also includes: **SCRIPTITE 50** *for unsurpassed printability and improved surface characteristics on boxboard.* **SCRIPTITE 33** *a melamine wet-strength resin.* **SCRIPTITE 52** *in combination with formaldehyde to give water resistance to folding boxboard and to jute liner.* **SCRIPTITE 54** *for outstanding*

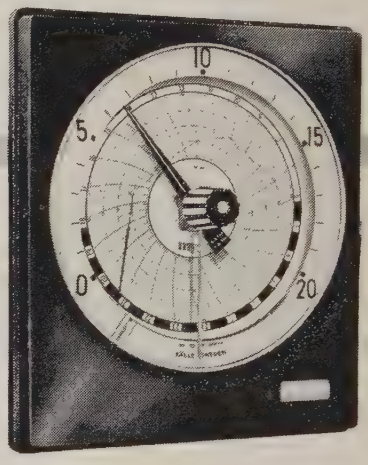
*SCRIPTITE: Reg. U. S. Pat. Off.

KÄLLE controllers and instruments

for outstanding reliable service

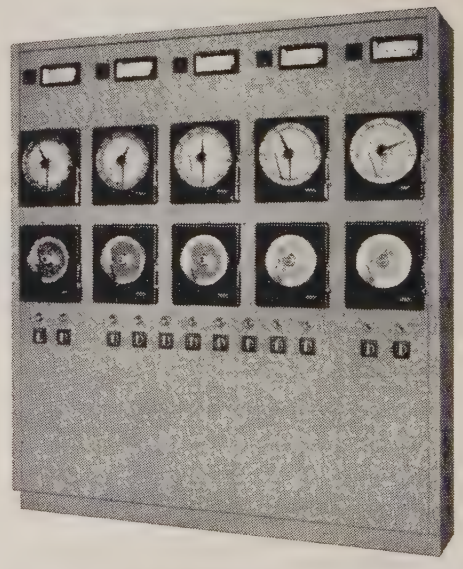


- The Källe controller Type Z60 is an "air-oil" operated control device.
- The controller itself generates easy moving, low pressure air for the impulse transmission.
- Hydraulic positioning assures distinct positioning forces.
- Air-oil combination yields instantaneous 1:500,000 impulse-positioning force ratio.
- PID-action for rapid response, perfect positioning and hunt free control.



Thermoelectric indicator and recorder. Derivative action for quick, distinct pointer movement. Also, three series of small instruments for panel mounting.

Centralized programmed control panel for sulphite and sulphate cooking.



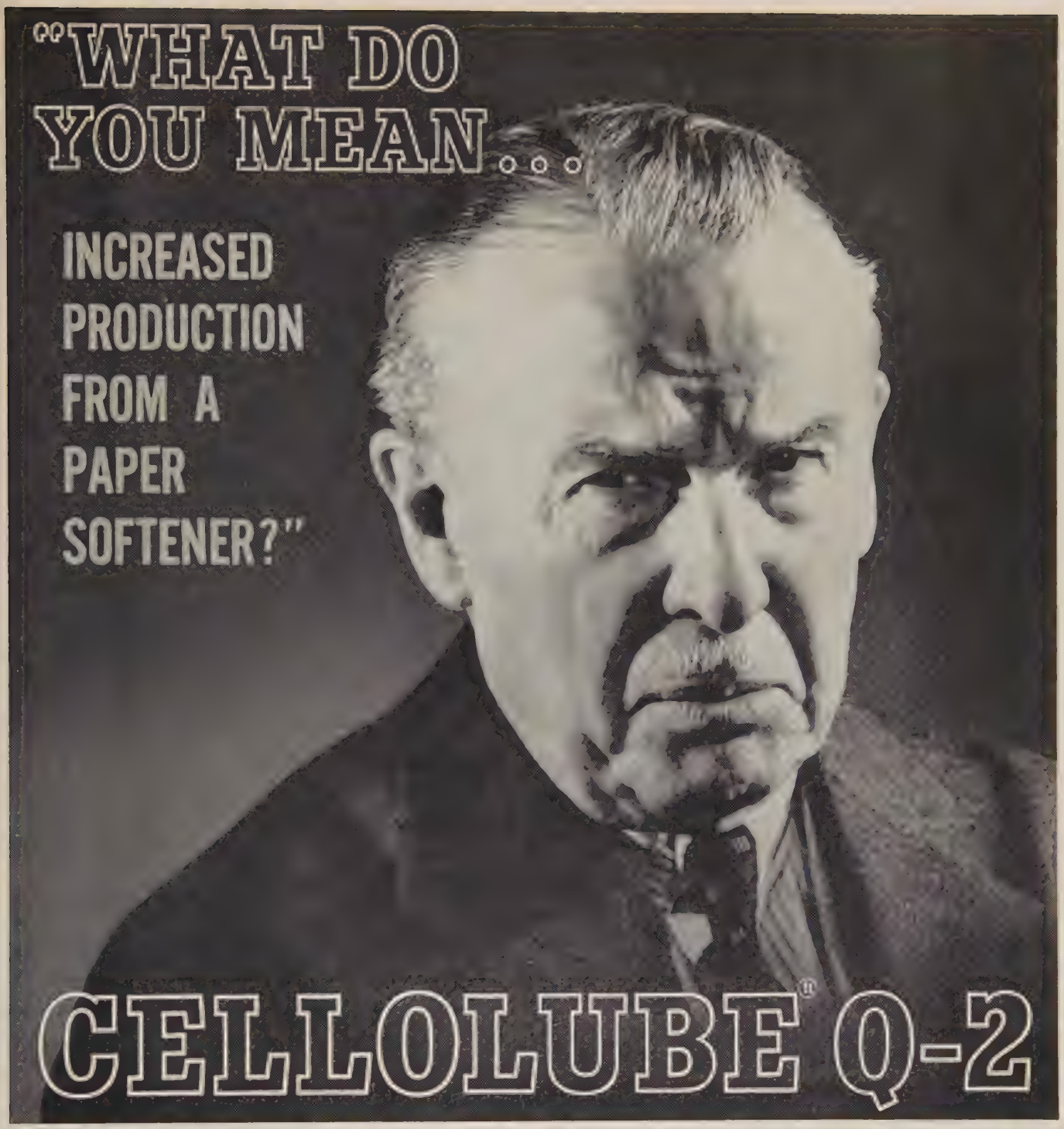
OUR FORTY YEARS OF EXPERIENCE

Forty years of Källe experience is at your service. Whether you need an individual instrument or a complete control system, you can rely on Källe equipment for product quality, accurate operation, and minimum maintenance.

Our experience in the control field is extensive. It includes the following operations in pulp and papermaking:

centralized programmed cooking
complete feeder control apparatus for pulp stock, dyes, and other additives
control of vacuum in suction boxes and press portion
dry-end steam pressure control and condensate removal
flow and level regulation
dew-point and draft control in recovery unit
instrument panels and complete remote control equipment are also available.

AKTIEBOLAGET KÄLLE-REGULATORER
SÄFFLE-SWEDEN



Quaternary Ammonium Paper Softener

We mean what we said . . . *and more!*

For Cellolube Q-2 not only gives a softer, more "sellable" sheet, but it boosts production and cuts production costs as well!

A few seconds after Cellolube Q-2 is added, its unique lubricating action goes to work . . . and *paper output increases from 10 to 20%*, due to stepped-up machine speed. And when speed is electronically controlled, production stays constant but *power demand drops.*

Along with stepped-up production and current-cost economies, Cellolube Q-2 increases doctor blade life up to 40% . . . releases sheets without slipping or sliding . . . and eliminates picking and sticking. As a result, operators have more time to spend on quality . . . and you get a better, softer, more economical sheet than ever before!

Like to *see* what we mean? We're prepared to demonstrate Cellolube Q-2 in your own mill, on a full-scale trial.

For details write, wire, phone:

TANATEX CHEMICAL CORPORATION

Belleville Turnpike, Kearny, New Jersey
WYman 8-0732

ALSO

Tanatex Chemical (Holland) N.V.
Der Kinderenlaan 3
Laren N.H., Holland

Chemtex Products Limited
49 Densley Avenue
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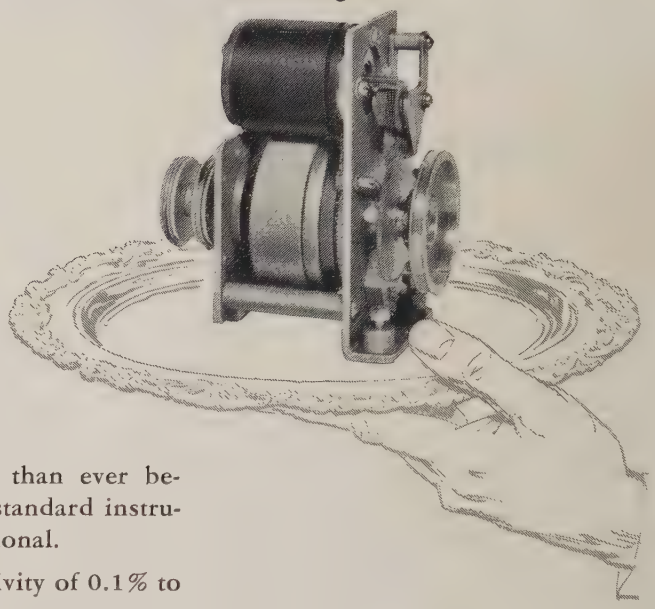
CHEMICALS FOR DIFFICULT PROCESSING

What makes **TRANSCOPE®** *Electronic and Pneumatic Recorders*

the most wanted miniature instruments on the market today?

The answer is Taylor SERVO POWER



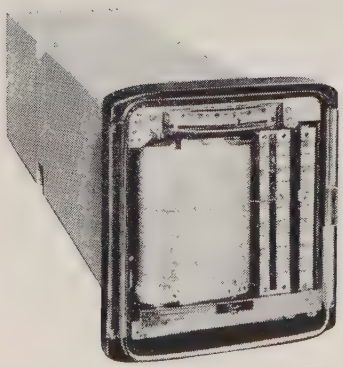
In the past 2 years thousands of Servomatic motors have proven the inherent advantages of this Taylor innovation. Heart of the 90J (pneumatic) and 700J (electronic) recorders, it puts TRANSCOPE Recorders in a class by themselves. It is the most reliable device yet developed for converting an input signal into an accurate record. The tremendous power of these Servomatic motors—150 times greater than the bellows type (pneumatic), over 1,000 times greater than galvanometer systems (electronic)—gives you all these advantages:

- Greater accuracy than ever before—½ of 1% in standard instruments, ¼ of 1% optional.
- Threshold sensitivity of 0.1% to input signal.
- 3 months' ink supply mounted on the pen—no troublesome capillary.
- Lifetime recording accuracy—no critical pen pressure.
- No flimsy linkages and levers.
- Truly rectilinear chart—no curved time lines.

Plus these optional features: alarm contacts—retransmitting potentiometer—function generation—digital output with encoder discs.

* * *

Ask your Taylor Field Engineer for full details of all the many superior features of TRANSCOPE servo-operated recorders. Or write for **Bulletin 98286** (pneumatic) or **98335** (electronic). Taylor Instrument Companies, Rochester, New York, or Toronto, Ontario.



Other TRANSCOPE advantages:

- Unconfused readability—because chart record, pen and set-point are side-by-side.
- 4" chart reads from left to right—on rectilinear coordinates.
- Controller response adjustments may be made from the front—you can watch the results.
- Separate Plug-in Set Point Transmitters permit staying on automatic control—even when recorder slide is removed.
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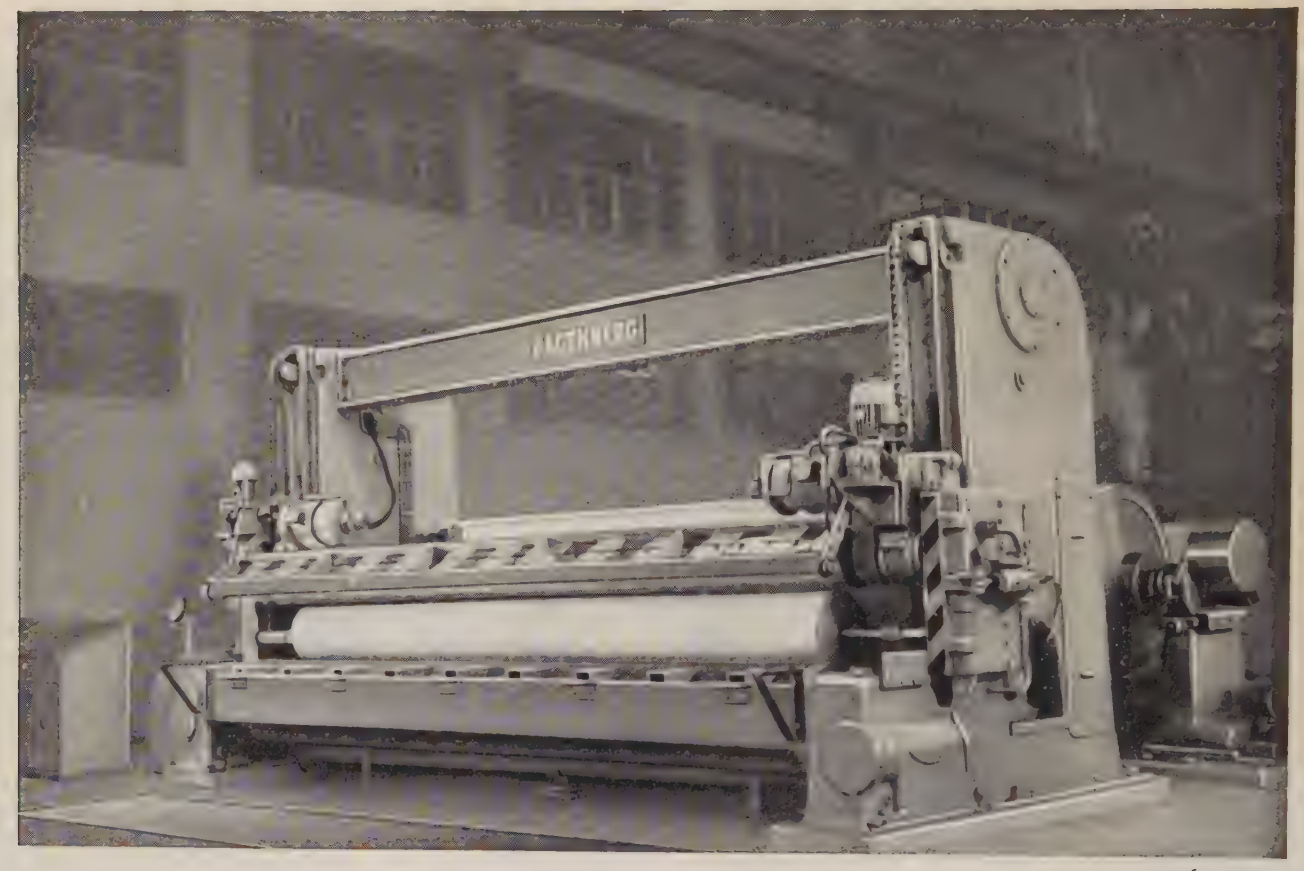

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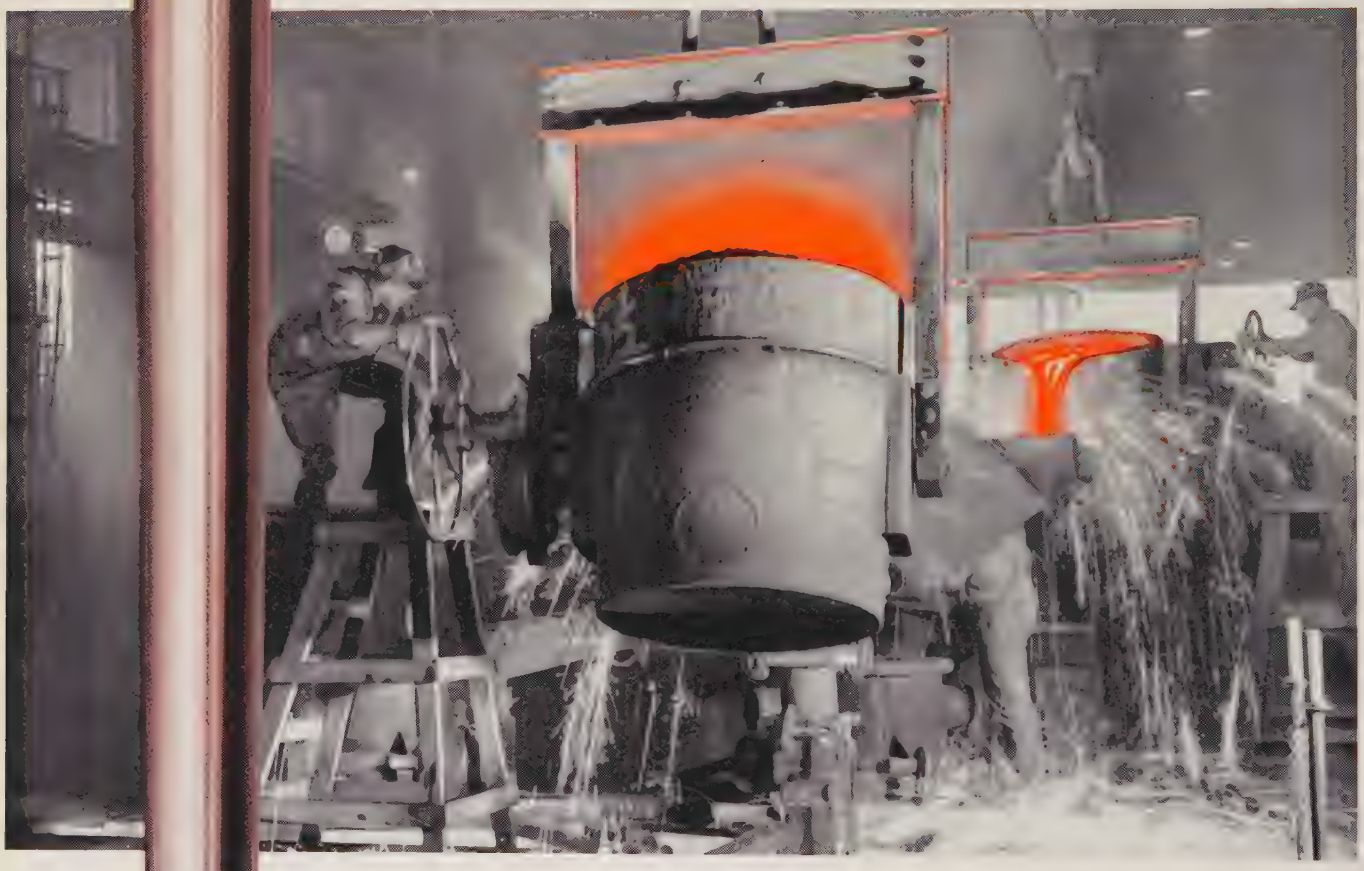

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Application of Diffusion Theory to the Washing of Kraft Cooked Wood Chips

SAMUEL W. McKIBBINS

The principles of diffusion theory are applied to the washing of kraft cooked wood chips. Data were obtained for extraction in the transverse and longitudinal directions of the wood for several temperatures and sample thicknesses which correlated closely with the equations predicted by diffusion theory. The longitudinal diffusion coefficients obtained from these data were approximately two to three times as large as those in the transverse direction. Data were also obtained for the extraction of sodium from kraft cooked samples of rectangular blocks and conventional pulp chips in which multidirectional diffusion occurred. The data for the rectangular blocks correlated closely with the theoretical predictions, but the correlation for the actual pulp chips was only approximate due to variations in their physical dimensions and geometries.

THE first major unit operation in the recovery of chemicals in a kraft pulp mill is the washing of the pulp after the cooking cycle. This is usually accomplished in several stages on rotary vacuum washers in which the pulp moves countercurrently to the wash liquor. In this type of washing, the cellulose fibers are in the form of a mat and the major portion of the chemicals is removed by a physical displacement with the wash liquor. Another type of equipment which is also employed is the diffusion washer in which the pulp is contained in a large tower or tank and the wash liquor passes through it. In this process, a diffusion mechanism probably controls the removal of a large portion of the chemicals from the matted fibers.

In both of these washing operations, the cellulose fibers are no longer in their original chip form but have been reduced to a pulp by some type of "blowing" operation. It is also of interest, however, to establish the desirability of washing pulp which is maintained in its original chip structure. The feasibility of washing pulp in this form is determined to a large extent, of course, by the efficiency and rates of removal of the chemicals from this structure. This paper reports the results of an investigation to measure these rates experimentally over a range of variables and to relate them to the washing process.

It seemed quite probable at the outset that the rate of removal of chemicals from a chip structure would be controlled by a diffusion mechanism. Although liquid phase diffusion through porous media is normally a very slow phenomenon, it was felt that after the cooking operation the chip structure would be sufficiently changed to offer little resistance to mass transfer. A search of the literature revealed that considerable work has been done in measuring the rates of diffusion of various materials through uncooked samples of wood. The particular purpose of most of these investigations was to elucidate the mechanism and rates of transport of cooking liquors into wood chips in order to optimize the cooking operation. There appeared to be little information available, however, on the change in rates after the wood has reacted to some degree with the cooking chemicals. The major distinction of this investigation, then, is that it is concerned with diffusion in wood which has received a high degree of change from its natural state by means of a conventional kraft cooking operation. Specifically, this investigation was concerned with the extraction of the sodium ion since it is this ion which is of prime importance in the kraft recovery process.

SAMUEL W. MCKIBBINS, Chemical Engineer, Kimberly-Clark Corp. Neenah, Wis.

NOMENCLATURE

A	=	frequency factor, sq. cm. per sec.
c	=	concentration, g. per cc.
D	=	diffusion coefficient, sq. cm. per sec.
exp	=	exponential
E	=	activation energy, g. cal./g. mole
L	=	thickness or length, cm.
R	=	gas constant = 1.987 g. cal./°K. × g. mole
t	=	temperature, °F.
T	=	absolute temperature, °K.
x	=	distance, cm.
Y	=	$(c - c_o)/(c_i - c_o)$, fraction of unextracted material
θ	=	time, sec.

Subscripts

a	=	average
i	=	initial
o	=	outside
x, y, z	=	coordinate directions

THEORETICAL BACKGROUND

The method of washing described above is really a type of liquid-solid extraction in which water is the solvent. It has been found in the past that many extractions of this type are controlled by diffusion mechanisms and hence the well-known integrated forms of the diffusion equations could be used to correlate the data. When the limitations inherent in these equations are considered, however, it is somewhat surprising that they apply to a porous material of such heterogeneity as wood. Osburn (*1*) has demonstrated that deviations can be expected when the material contains capillaries of various sizes and angles to the exposed faces or capillaries that lead from reservoirs. These conditions certainly exist in wood as do the additional deviations from homogeneity such as variations in density, moisture content, and structural components. The fact that the equations do apply in some cases, would seem to indicate that some averaging phenomenon balances the natural variations.

Unsteady-state unidirectional and isothermal diffusion with a constant diffusion coefficient may be described by Fick's second law of diffusion in this form:

$$\frac{\partial c}{\partial \theta} = D\left(\frac{\partial^2 c}{\partial x^2}\right) \qquad (1)$$

homogeneous material of constant width L which has a uniform initial concentration c_i, and that the solute leaves at both faces which are maintained at a constant concentration, c_o the following initial condition and boundary conditions apply:

Initial condition: $\theta = 0$, $c = c_i$ for all x
Boundary condition: $x = 0, L$, $c = c_o$ for $\theta > 0$

Equation (1) may then be integrated to yield the following expression for the average concentration as a function of time:

$$Y_{ax} = \left(\frac{c_a - c_o}{c_i - c_o}\right) = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp\left[\frac{-(2n-1)^2\pi^2 D\theta}{L^2}\right] \quad (2)$$

It will be noted that this infinite series solution reduces to one term for values of $(D\theta/L^2)$ greater than 0.03 (i.e., Y_{ax} less than 0.6). This allows the experimental determination of D from the slope of the line obtained by plotting ln Y_{ax} versus θ.

In those instances where diffusion occurs in more than one direction, equation (1) must be expanded to include the new coordinates and diffusion coefficients involved. The solution of these types of second order partial differential equations is not as straightforward as that for unidirectional diffusion and hence analytic solutions are rare. Newman (*2*) has shown, however, that for certain geometries and sets of boundary conditions, the solution for multidirectional diffusion is the product of the solutions for unidirectional diffusion for each of the coordinates involved. That is, the average concentration of a rectangular parallelepiped with diffusion in the x, y, and z directions will be equal to the product of the average concentrations obtained for each of these directions, namely:

$$Y_a = Y_{ax}\, Y_{ay}\, Y_{az} \quad (3)$$

equations (2) and (3) will be employed in the correlation of the experimental data given below.

EXPERIMENTAL TECHNIQUE

Three distinct types of experiments were performed which are summarized below and in Fig. 1.

1. Diffusional rates were measured in only the longitudinal direction and also only in the transverse direction. Longitudinal is defined here as parallel to the wood capillaries and transverse as perpendicular to them. For these experiments, blocks were cut from pulping logs of a Lake States spruce which were 1 by 1 by 0.25 in. and 1 by 1 by 0.125 in. Diffusion was restricted to the shortest dimension by coating the edges of the blocks with a Pliolite cyclized rubber compound after cooking. The open faces represented then either transverse or longitudinal surfaces.

2. Tridimensional diffusion was measured in blocks similar to those described above. In this case, however, the capillaries always ran parallel to the 1 by 1-in. face so that diffusion in the shortest direction was transverse diffusion as indicated in Fig. 1.

3. Diffusion rates were measured using regularly prepared wood chips from the same wood described above. These chips were carefully screened to obtain samples of approximately the same dimensions. The 45° angle and average dimensions of 1 by 0.6 by 0.115 in. as shown in Fig. 1.

These wood chips and blocks were cooked in a pilot plant digester under typical kraft cooking conditions which yielded permanganate numbers for the several batches varying from 13.4 to 26.7. This wide range resulted from having chips of various dimensions together in the same cook. Although the sodium concentration of the individual chips varied somewhat from one cook to another, the variation of the diffusion coefficient with concentration over the range involved is small and was not detected in these trials. After cooking, the chips were stored under constant high humidity conditions until the washing trials.

Of the many materials present in the cooked wood,

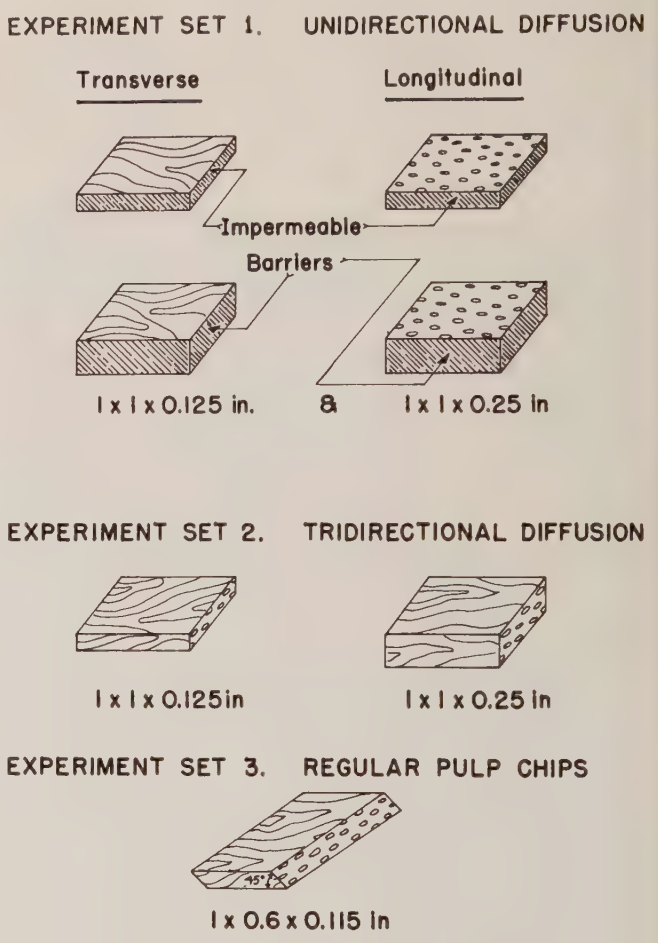

Fig. 1. Summation of geometries used in washing trials

the sodium ion is the most important from a recovery standpoint. It was decided, therefore, to measure its extraction rate from the cooked wood samples. The technique employed to measure the sodium content of a cooked wood chip involved a wet oxidation of the chip with gently boiling nitric acid until the solution was clear followed by a treatment with warm perchloric acid. The cooled solution was then diluted to a predetermined volume with distilled water and the sodium concentration measured by means of its emission spectra in a flame spectrophotometer.

Before each washing trial, each chip involved was weighed and the sodium content of several determined

to serve as a blank. In the unidirectional diffusion trials, the Pliolite rubber film was then applied. The chips were then placed in marked compartments of a wire basket which was submerged into a well agitated constant temperature water bath. The bath was of sufficient size to ensure that its sodium concentration remained essentially zero and trials were also run at various degrees of agitation to ensure that there was a negligible resistance to mass transfer in the water at the surface of the chips. At appropriate time intervals several chips were removed and placed in wide mouth flasks. When the last chips had been removed, they were all analyzed for their residual sodium content.

It had been shown that the ratio of the weight of sodium to the weight of the total chip was essentially constant for any given cook and constant humidity

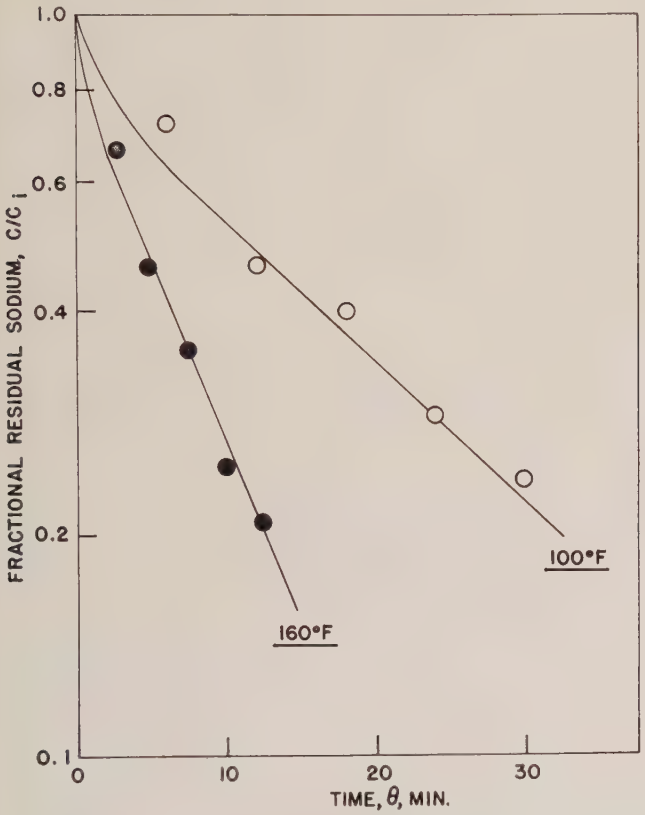

Fig. 2. Comparison of experimental results and theory for unidirectional longitudinal diffusion from 0.125-in. thick blocks

storage conditions. Therefore, from the sodium concentration of the blanks and the weight of each chip, it was possible to calculate the fractional residual sodium content of each chip (i.e., the weight ratio of sodium present after washing to that initially present).

RESULTS

In the unidirectional diffusion trials, the logarithm of the fractional residual sodium content was plotted versus the washing time as typified in Fig. 2, and from the slope of the straight line portion of these curves the values of the diffusion coefficients were determined. A linear regression analysis of the data was made employing the method of least squares and in all cases the correlation coefficients indicated a correlation significant at the 5% level. These diffusion coefficients for several temperatures, thicknesses, and directions are summarized in Table I. It appears that the coefficients are independent of path length L for transverse diffusion, but that in longitudinal diffusion they increase as the path length increases.

Since mass transfer by diffusion is a rate phenomenon, Glasstone, Laidler, and Eyring (*3*) point out that there will be an associated activation energy E which is required to elevate the diffusing molecules to that energy level sufficient to initiate molecular transport. Application of their theory of rate processes indicates that the diffusion coefficient may be related to the absolute temperature in the following manner:

$$D = A\sqrt{T}\exp(-E/RT) \qquad (4)$$

This equation indicates that a plot of the logarithm of $D/\sqrt{T}$ versus the reciprocal of the absolute temperature should result in a straight line with a slope de-

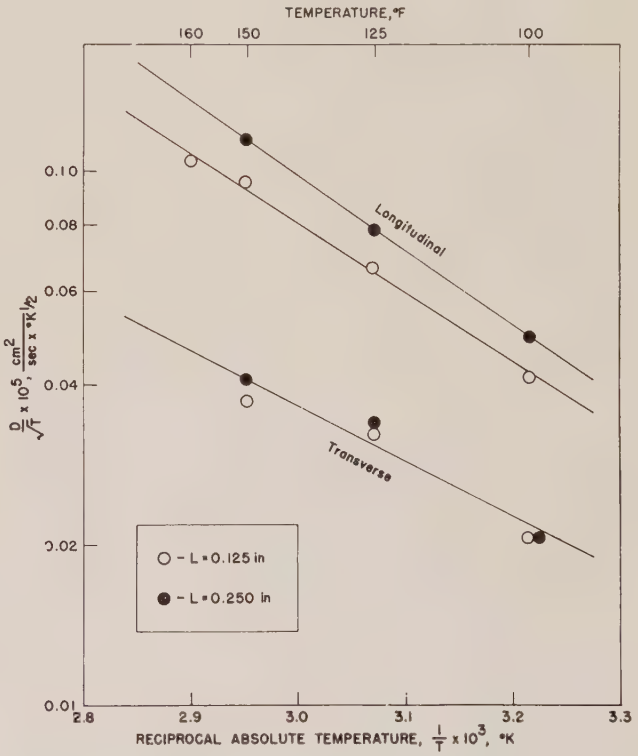

Fig. 3. Correlation of experimental longitudinal and transverse diffusion coefficients with temperature

pendent on the activation energy. Equation (4) was used to correlate the present data as shown plotted in Fig. 3. The particular frequency factors A and activation energies obtained from these plots are listed in Table I. Correlations were obtained for the 0.125 and 0.25 in. longitudinal coefficients individually and collectively for averages of the 0.125 and 0.25 in transverse coefficients.

In Fig. 4 is a comparison between theory and practice for tridirectional diffusion. The data points are from experiments with 1 by 1 by 0.125-in. wood blocks, and the theoretical curves were obtained by employing equation (3) and the diffusion coefficients of Table I. The coefficient for longitudinal diffusion in the one-inch direction was taken as that for a 0.25-in. length. Although the coefficient appears to be a function of length, the error involved is not more than a few per cent since diffusion in this direction accounted for

relatively little of the total mass transfer. The data for the 1 by 1 by 0.25-in. blocks correlated in a similar manner.

The data from the washing tests with regular pulp chips are summarized in Fig. 5. Since the chips are quite thin, it was felt that the diffusion would occur predominantly in one direction. Hence, the data are

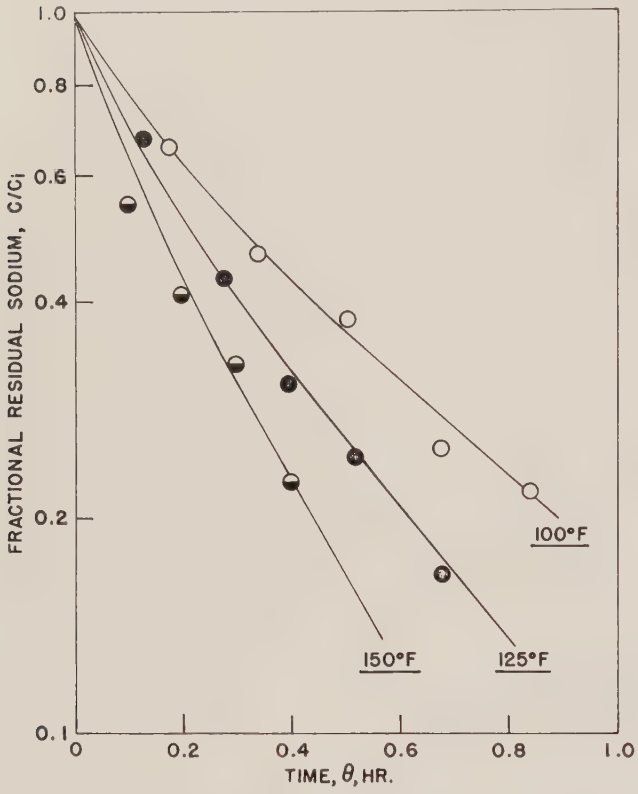

Fig. 4. Comparison of experimental results and theory for tridirectional diffusion from 1 × 1 × 0.125 in. wood block

plotted in Fig. 5 as a function of the dimensionless group $(D_x\theta/L^2_x)$ where D_x is the transverse diffusion coefficient from Table I and L_x is the average chip thickness, 0.115 in. The dashed line indicates unidirectional diffusion and the solid line is the theoretical curve for tridirectional diffusion from the rectangular parallelepiped indicated in the figure which approximates an actual chip. That is, its dimensions are 1 by 0.6 by 0.115 in., the capillaries are orientated as shown in Fig. 1, and the longitudinal diffusion coefficient is three times as large as the transverse diffusion coefficient.

DISCUSSION OF RESULTS

The close agreement of the experimental data and the theoretical curves indicates that diffusion theory can be successfully applied to the washing of sodium from kraft cooked chips. Despite the heterogeneous character of wood and the complex equilibrium of the sodium ions with the diverse anions the overall rate controlling mechanism, nevertheless, appears to be that of diffusion.

It is difficult to ascertain in what manner the various anions are associated with the movement of the sodium ion. There obviously is an equal transport of negative ions to maintain an electrical balance, but one can

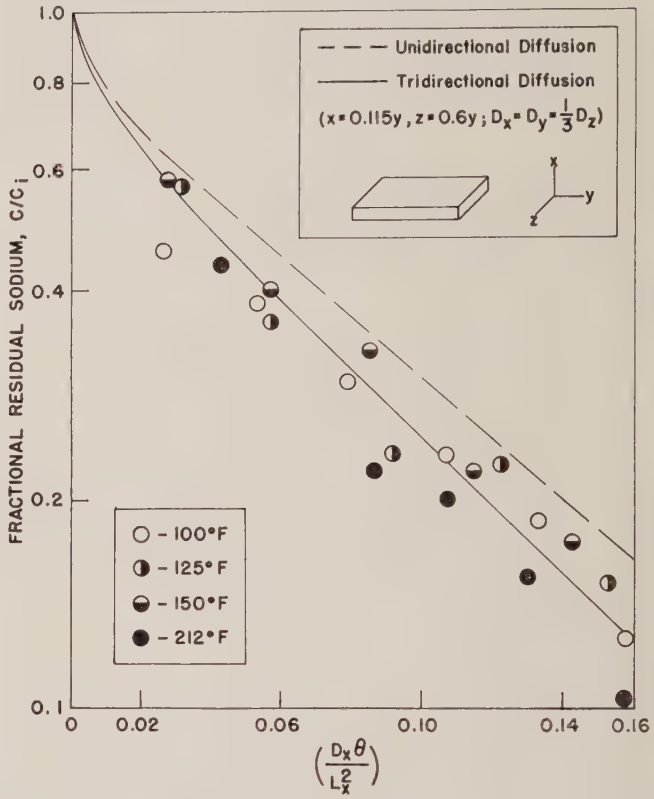

Fig. 5. Correlation of experimental results for diffusion from conventional pulp chips with theory for unidirectional diffusion and for tridirectional diffusion from a dimensionally similar block

only speculate concerning their relationships. It seems likely, however, that the list of anions should include hydroxyl, sulfate, acetate, formate, and other complex organic salts of the saccharinic acids and the thiolignin groups. The sodium salts of the first four ions mentioned diffuse in pure water at rates only slightly higher than those indicated in this work. Sodium hydroxide is the most mobile and has a diffusion coefficient approximately three and a half times that shown for longitudinal diffusion in Table I while sodium sulfate diffuses approximately twice as fast. Since it is probable that the larger organic molecules mentioned diffuse more slowly and would consequently reduce the overall rate, it appears that the measured diffusion rate is close to the rate of the same combination of ions in water alone. That is, in a cooked chip the cellu-

Table I. Experimental Diffusion Coefficients and Temperature Correlation Constants

Direction of diffusion	*Diffusion length, L, in.*	*Diffusion coefficients, D × 10⁵, sq. cm. per sec. Temperature, t, °F.* 100	125	150	160	*Frequency factor A × 10³ sq. cm. per sec.*	*Activation energy, E, cal./g. mole*
Longitudinal	0.125	0.71	1.2	1.8	1.9	8.92	6170
	0.250	0.84	1.4	2.1	...	20.2	6580
Transverse	0.125	0.36	0.58	0.68	...		
	0.250	0.36	0.60	0.75	...	0.564	4870

lose structure presents relatively little resistance to mass transfer. Thus it may be considered as a type of "hindered diffusion" in which the wood structure acts only as a discontinuous barrier to the movement of the ions through the water phase contained in the structure. This picture is consistent with the magnitude of the activation energies found in this work which are quite similar to those for most sodium salts in water alone.

As would be expected, the rate of movement in the transverse direction was somewhat lower due to the hindrance offered by the tracheid walls. From the values listed in Table I, it will be noted that the ratio of the coefficients for longitudinal to transverse diffusion vary from approximately two to three. It would be expected that this ratio would be lower in cooked wood than in uncooked wood since a relatively greater change in the structure occurs in the transverse direction. With the solubilizing of the middle lamella which joins the various cellulose tubes the resistance to mass movement has been considerably reduced. There is no such comparable change in the longitudinal direction.

The work of previous investigators confirms this. In a study of the diffusion of sodium chloride through various woods, Behr, Briggs, and Kaufert (*4*) found that the ratio of coefficients for longitudinal to transverse diffusion was approximately 40 for uncooked spruce. Their value for longitudinal diffusion of 0.8 by 10^{-5} sq. cm. per sec. at 20°C. was higher than that found in this work (which is probably due to the higher mobility of sodium chloride over the combination measured here), but their value of 0.020×10^{-5} sq. cm. per sec. for transverse movement was considerably less. Yorston (*5*) recorded similar values for potassium chloride into uncooked black spruce with a ratio of about 33. Moreover, he showed that this ratio dropped to about 5.8 after the samples had been heated in a sulfite liquor at 110°C. Christensen (*6*) also reports a ratio of about 40 for the diffusion of sodium chloride in uncooked pine samples. His values for longitudinal diffusion which vary from 0.5×10^{-5} to 1.06×10^{-5} sq. cm. per sec. at 20°C. and 50°C. compare with the values reported here as do the values for activation energies. Comparison of absolute values is difficult, however, due to changes in the many variables such as density and species.

It was found that the activation energy for diffusion in the longitudinal direction was higher than that in the transverse direction. Again the explanation probably lies in the structure of the wood. Longitudinal diffusion is essentially through long capillaries while transverse diffusion is essentially between the tracheid tubes. Christensen (*6*) points out that in capillary diffusion the activation energy is in an inverse proportion to the capillary diameter. This would explain why the relatively less restricted transverse diffusional paths would require a low activation energy. Table I also indicates a difference for the two longitudinal activation energies. With the few data points used to determine the values involved, however, it is difficult to state whether the difference is real or due to experimental error.

The fact that the longitudinal diffusion coefficients are larger for the thicker chips is not consistent with diffusion theory. Since they were larger in every case, as opposed to the transverse coefficients, they are reported distinctly. Christensen and Williams (*7*) have reported the same phenomenon and suggest that the area under the impermeable barrier, which is larger in thicker samples, contributes to mass transfer.

In all the trials, the chips were initially at room temperature when they were placed in the water bath. This implies the existence of a short transition period during which the extraction occurs under nonisothermal conditions. Calculations using an assumed thermal diffusivity for the wet wood indicated, however, that this period is negligibly short. In any case, this should not materially change the value of the diffusion coefficient since this transition period would not greatly affect the slope of the extraction curve but would only serve to displace it horizontally.

The application of diffusion theory to predict the sodium content was rather successful for the uniform geometry shown in Fig. 4. The wide variation in geometries and sizes in regular pulp chips, however, precludes any similar degree of success for these chips. The fact that they correlated as well as is shown in Fig. 5 is probably due to the care taken in carefully preselecting chips of uniform size prior to washing. Because a regular chip is quite thin, the diffusion process approaches that of unidirectional diffusion as is shown in Fig. 5 by the dashed line, but due to the longitudinal transport which is associated with a larger coefficient, the data lie somewhat below it.

The solid line in Fig. 5 does indicate, however, that the sodium content can be approximated by considering a regular geometry of similar proportions. This curve is not restricted to any absolute size, but only to the physical proportions and ratios of diffusion coefficients indicated. Since most wood chips are approximated by these restrictions, this curve may apply to most extractions regardless of the actual chip sizes. If there is a wide distribution of sizes, however, it may be necessary to divide the total sample into groups of characteristic length L and apply the curve of Fig. 5 individually to each group. The total fraction extracted is then equal to the sum of that for each group multiplied by the fraction of the total extractable material which it represents. This procedure has been described in detail by Osburn (*1*). The theoretical rationalization of this practice of applying a geometric substitute is perhaps somewhat tenuous, but due to the irregularities of the material being considered and the consequent accuracy of any experimental data, it is probably justified.

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The author is indebted to D. J. Casanova, J. A. Gonzales, and The Institute of Paper Chemistry for collecting and analyzing the experimental data.

C. J. West Memorial paper presented at the meeting of the Lake States Section of TAPPI held in Neenah, Wis.

Peroxide Bleaching of Kraft Pulps

NILS HARTLER, ENAR LINDAHL, CARL-GEORG MOBERG, and LENNART STOCKMAN

The stability of alkaline solutions of hydrogen peroxide is shown to be influenced by the material of which the reaction bottle is made. Certain metal ions impair the stability. Besides magnesium sulfate and silicate, chelating substances (e.g., EDTA) have a stabilizing action. For successful stabilization amounts sufficient to chelate all metal ions have to be added. In final stage bleaching of sulfate pulps, hydrogen peroxide produces a pulp of maximum brightness when applied at a pH of approximately 11. The amount of residual hydrogen peroxide must not fall below a certain minimum value in order to attain optimal effect. When a sufficient amount of silicate is present as stabilizer a surprisingly low percentage of the added peroxide is consumed. A high pulp consistency and a high temperature are favorable to bleaching. The most attractive feature of a peroxide-bleached pulp is its low brightness reversion.

Because of the high price of peroxide its use for bleaching chemical pulps is not so widespread. For sulfate pulps the hydrogen peroxide can be added either in an alkaline extraction stage or it can be used in a separate final bleaching stage.

There are undoubtedly many advantages to be gained by using hydrogen peroxide but one of the problems that arise is the instability of peroxide solutions. The first part of this investigation was, therefore, devoted to a study of the stability of dilute alkaline hydrogen peroxide solutions while in the second part an examination was made of hydrogen peroxide bleaching of a prebleached sulfate pulp. The bleaching agent competitive with hydrogen peroxide is chlorine dioxide and, therefore, a comparison was made of the bleaching action of hydrogen peroxide and of chlorine dioxide.

STUDIES ON THE STABILITY

Stabilities of Aqueous Hydrogen Peroxide Solutions

Hydrogen peroxide is a weak acid with a dissociation constant of approximately 10^{-11} at 60°C. This means that at a pH <9 it is only negligibly dissociated. As can be seen from Fig. 1 the dissociation increases with pH from 9 to 13. This figure also shows the influence of temperature on the dissociation.

Dissociation with the formation of the ion HO_2^- is a prime requisite for any appreciable bleaching action by hydrogen peroxide on chemical pulps. Thus when considering the stability of an aqueous solution of hydrogen peroxide for pulp bleaching it is exclusively the pH interval 9 to 13 that is of interest. This pH interval coincides with the pH interval of instability; below pH 9 such a solution is practically stable. This strongly indicates that the ion HO_2^- is involved in both the bleaching action and the decomposition.

The decomposition was studied in the following way:

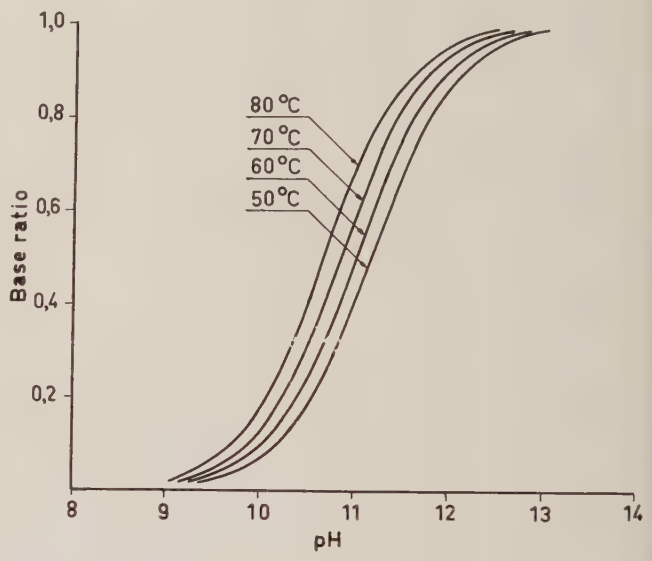

Fig. 1. Dissociation of hydrogen peroxide with pH

Solutions containing 1.3 g.p.l. of hydrogen peroxide and varying amounts of metal ions and stabilizers were kept in a thermostated water bath at the desired temperature. Samples were withdrawn at intervals and the amount of hydrogen peroxide was determined by titration with 0.1 *N* sodium thiosulfate solution. The pH was regulated by the addition of sodium hydroxide and was determined at the beginning and at the end of the experiment. The difference between the initial and the final pH was usually 0.4 units or less, except at the very lowest pH values and in those experiments where a high percentage of the hydrogen peroxide was decomposed. In these two cases the difference was somewhat larger. The hydrogen per-

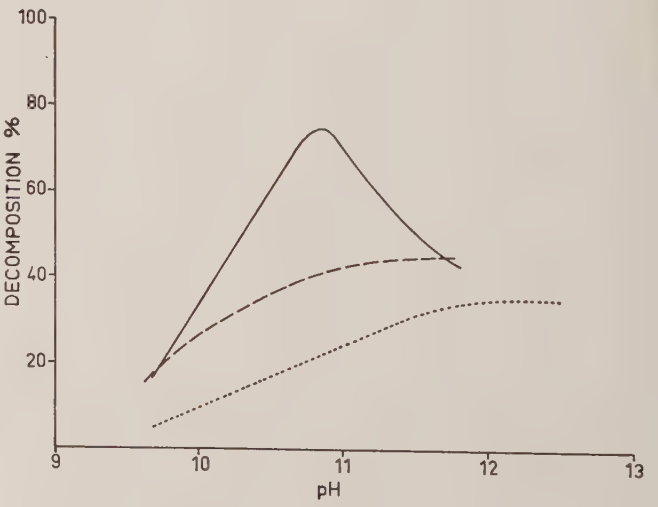

Fig. 2. Decomposition of hydrogen peroxide solution (1.3 g./l.) at 60°C. for 3 hr.

—— Glass; – – – – dichromate-washed glass; . . . . . polyethylene

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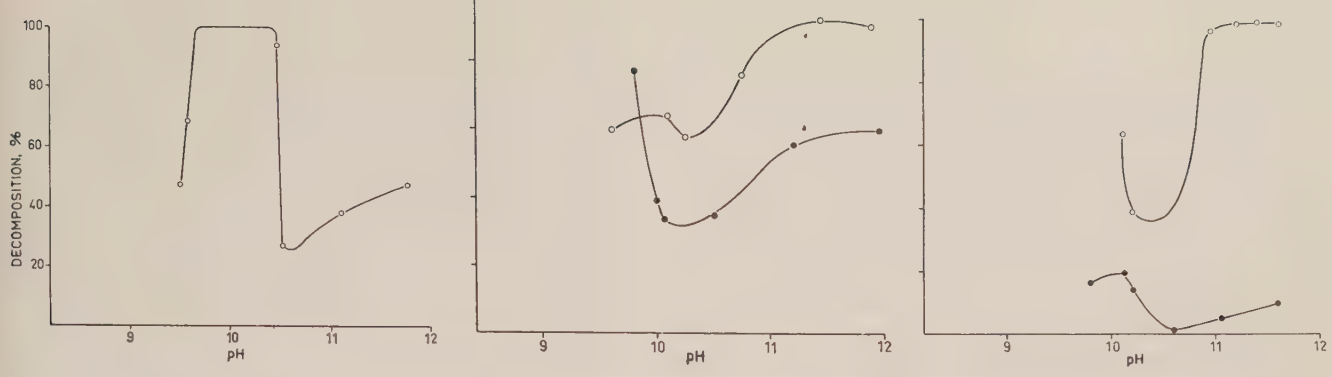

Figs. 3, 4, and 5. Decomposition of hydrogen peroxide solutions (1.3 g./l.) containing different metal ions

Fig. 3. 0.2 mg./l. Mn^{++}; 1 hr. at 60°C. Fig. 4. ○ 0.2 mg./l. Mn^{++} + 78.5 mg./l. Ca^{++}; ● 0.2 mg./l. Mn^{++} + 15.7 mg./l. Ca^{++}; 1 hr. at 60°C. Fig. 5. ○ 0.2 mg./l. Mn^{++} + 0.05% $MgSO_4$; ● 0.05% $MgSO_4$; 3 hr. at 60°C.

oxide used was of analytical grade. The amount of added stabilizer is given in percentage of an imaginary pulp quantity assuming 8% consistency, and the amount of added metal ions in percentage of the total amount of liquor.

The material of which the reaction bottle was made had a decisive effect, especially in the case of pure hydrogen peroxide solutions. This effect is shown in Fig. 2 where the percentage of hydrogen peroxide decomposed is plotted against the pH for glass, dichromate-washed glass and polyethylene bottles. It seems probable that the decomposition is catalyzed by small quantities of impurities present on the surface of the glass.

According to Erdey (*1*) the main decomposition reaction should be a reaction between the HO_2^- ion and undissociated hydrogen peroxide according to the equation.

$$H_2O_2 + HO_2^- = H_2O + OH^- + O_2$$

According to this equation the maximum decomposition could be expected at 50% dissociation, that is, when the pH is equal to the pK for the dissociation reaction. Since the pK is approximately 11 at 60°C. the decomposition should have a maximum at pH 11 if the decomposition proceeds according to the above reaction. As can be seen in Fig. 2 no maximum is found when polyethylene bottles are used and it can be concluded, therefore, that the decomposition mechanism is not so simple as this. However, since the decomposition increases with increasing pH it may be concluded that the decomposition is closely associated with the ion HO_2^- and also that the presence of this ion is necessary for the decomposition.

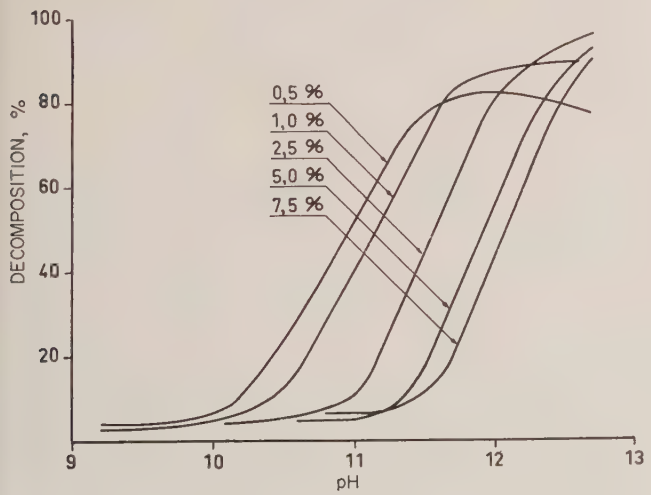

Fig. 6. Decomposition in 3 hr. at 60°C. of hydrogen peroxide solution containing sodium silicate

The amount of silicate is calculated on an imaginary pulp quantity at a concentration of 8%. When recalculated into g./l. the amount of silicate for the two extremes will be 0.44 and 6.6 g./l., respectively.

Effect of the Addition of Various Metal Ions

The addition of small amounts of metal ions to hydrogen peroxide solutions may give a slight improvement in stability as is the case with calcium ions and ferric ions below pH 11, or may make little difference, as for instance with lead ions and aluminum ions. Of great interest in bleaching are the metal ions which lower the stability, among which are manganese, ferric ions above pH 11 (*2*) and cupric ions.

The effect of the addition of manganese ions was studied by measurements of decomposition after a reaction time of one hour. As shown in Fig. 3 the stability is drastically reduced with increasing pH, up to approximately 9.5. Increasing the pH further gives an improvement in stability followed by a slight deterioration. At a pH of approximately 10.5 the manganese is precipitated as manganese hydroxide and this may be the reason why improved stability is found at this pH. The addition of both manganese and calcium ions seems to give an improved stability below pH 11 but poorer stability above pH 11, as is shown in Fig. 4. No reasonable explanation was found for this effect.

The addition of cupric ions impairs the stability of hydrogen peroxide solutions; their effect is less pronounced than that of manganese ions below pH 10.5, more pronounced above pH 10.5.

Effect of the Addition of Various Stabilizers

The addition of magnesium sulfate improves the stability of aqueous hydrogen peroxide solutions and a maximum of 20% decomposition was obtained over the pH interval 9–12. If manganese ions are also present magnesium sulfate improves the stability only within a very narrow pH range around approximately pH 10.5. As can be seen from Fig. 5, when manganese

ions are present there is 100% decomposition within 3 hr. at a pH above 11 even when magnesium sulfate is added.

The effect of the addition of sodium silicate to a solution of hydrogen peroxide is shown in Fig. 6. Regardless of the amount added and of the other components present, the addition of sodium silicate gives reasonable stability up to a certain pH but extremely poor stability at a pH somewhat higher. This limiting pH was approximately 11 when sodium silicate only was added to hydrogen peroxide solution, approximately 10.5 when manganese ions were also present and approximately 11.7 when sodium silicate, magnesium sulfate, and manganese ions were all present.

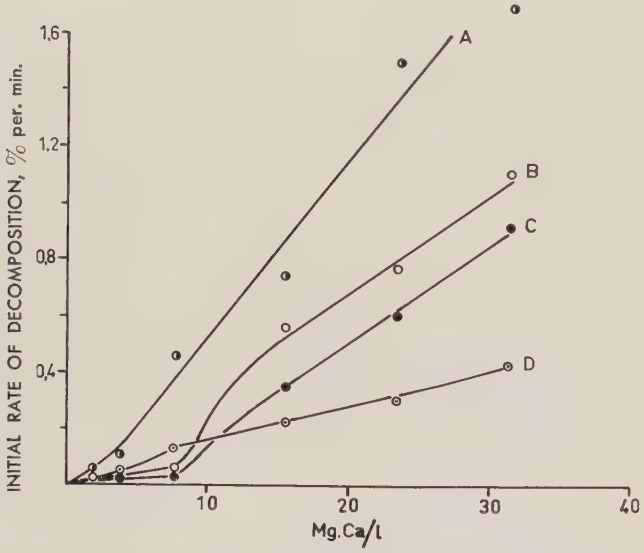

Fig. 7. Decomposition rate (% per min.) of hydrogen peroxide solutions (1.3 g./l.) with amount of calcium added at pH 11 and 60°C. in the presence of either EDTA or of polychelate and 0.2 mg./l. Mn^{++}

A, 0.05% EDTA; B, 0.10% EDTA; C, 0.15% EDTA; D, 0.1% polychelate.

The mechanism of sodium silicate stabilization can be explained by complex formation between hydrogen peroxide and free silicic acid. The following equilibria should be valid for the formation of the complex and for the dissociation of the hydrogen peroxide (X is the free silicic acid, K_1 is the complex formation constant, and K_2 the hydrogen peroxide dissociation constant).

$$H_2O_2 + X \rightleftharpoons H_2O_2X$$

$$\frac{[H_2O_2X]}{[H_2O_2]\ [X]} = K_1$$

$$H_2O_2 + H_2O \rightleftharpoons H_3O^+ + OOH^-$$

$$\frac{[H_3O^+]\ [OOH^-]}{[H_2O_2]} = K_2$$

and therefore:

$$[H_3O^+] = \frac{[H_2O_2X]}{[OOH^-]} \frac{K_2}{[X]\ K_1}$$

From this it may be concluded that for given sodium silicate and peroxide concentrations the concentration of HO_2^- is increased with increased pH which means poorer stability. On the other hand at a given pH the concentration of HO_2^- will be decreased as the amount of free silicic acid is increased which means improved stability. This is in accordance with the results of the experiments and the effect of sodium silicate may therefore be due to complex formation. This would influence the dissociation of the peroxide and thus would also affect the stability of the solution.

To obviate the reduction in stability it has been suggested that a chelating agent could be added to the hydrogen peroxide solution to chelate with any metal ions. Usually a number of metal ions are present in bleach liquor though probably only a few of them are harmful to the stability. It would be desirable, therefore, to have selective reagents chelating only the harmful metal ions so that the amount of reagent required is smaller and economically more reasonable.

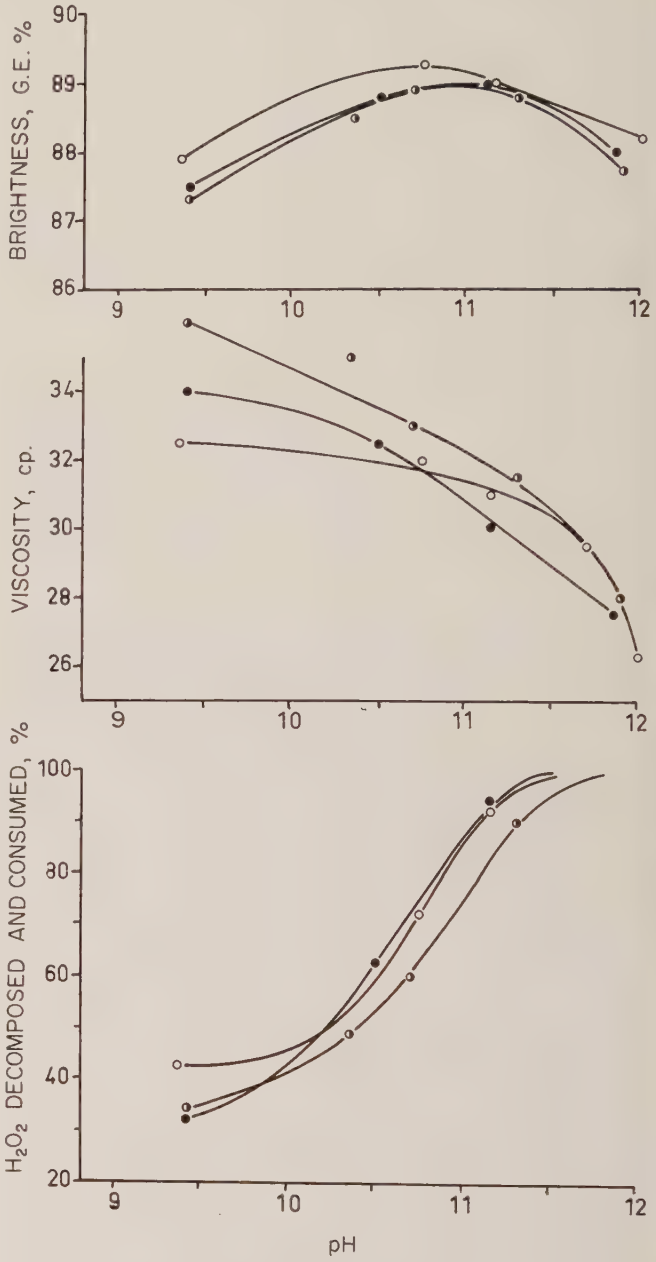

Fig. 8. Brightness, viscosity, and peroxide decomposition and consumption as a function of pH

○ no stabilizer; ● 0.05% EDTA; ◑ 0.05% $MgSO_4$. H_2O_2 added, 0.3%; pulp consistency, 6%; distilled water; 4 hr.; 70°C. Brightness of the pulp before the H_2O_2 stage, 81.6% G.E.; viscosity, 37 cp.

However, there are practically no chelating substances of this sort. Tests were made with ethylenediaminetetraacetic acid (EDTA) and with "Polychelate" (from AB Rexolinfabriken, Hälsingborg, Sweden). Working on the assumption that the two main metal ions present in a bleach liquor were calcium and manganese, some experiments were made with solutions containing different quantities of these two ions. It was found that a little more than the amount of chelating substances corresponding to the amount of calcium ion

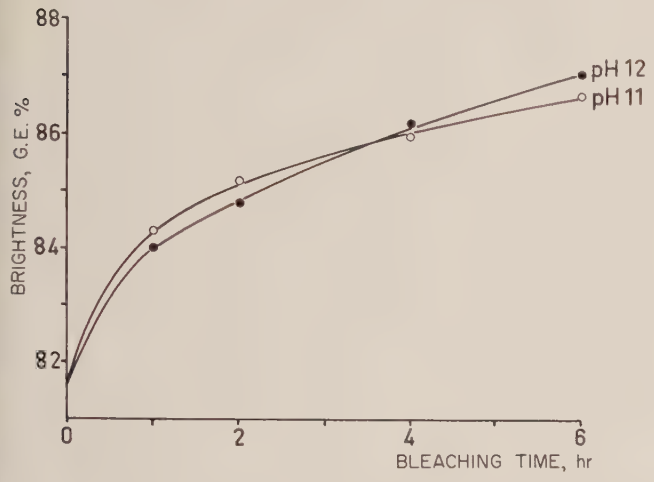

Fig. 9. Brightness of the pulp as a function of bleaching time at pH 11 and 12

Constant HO_2^- concentration; pulp consistency, 0.5%; distilled water; 70°C.

present had to be added in order to obtain any chelating action on the manganese ions and improvement in stability. The results are shown in Fig. 7. It can be seen that when there was a large amount of calcium ions present it was not possible to obtain good stability even after adding fairly large amounts of chelating substance. This means that if any substantial amount of calcium ions is present in the bleach liquor it is not economic to use nonspecific chelating substances to eliminate the deleterious effect of manganese ions on the stability of hydrogen peroxide solutions.

BLEACHING EXPERIMENTS

Procedure

Some bleaching experiments were made using peroxide as a final-stage bleaching agent for a prebleached softwood sulfate pulp. The pulp had a brightness of 80% G.E. and a viscosity of 40 cp. TAPPI. The bleachings were done in polyethylene bottles (100 g. pulp) using distilled water and chemicals of analytical grade. At the highest consistency, 14%, polyethylene bags (50 g. pulp) were used in order to facilitate mixing at bleaching temperature. The bleached samples were after-treated with 1% SO_2 water for 15 min.

The brightness sheets were prepared according to CCA 27:58 and their brightness determined in the Elrepho using filter R 46 T and reported as per cent G.E.

The viscosity was determined according to CCA 16 and the result converted to centipoise TAPPI.

Influence of pH

As previously mentioned there is no pronounced bleaching action below pH 9. As shown in Fig. 8 the brightness increases and the viscosity decreases with increase in pH up to approximately 11, other conditions being the same. At this pH there is still some hydrogen peroxide left but the amount remaining drops almost to nil if the pH is increased further. This is probably

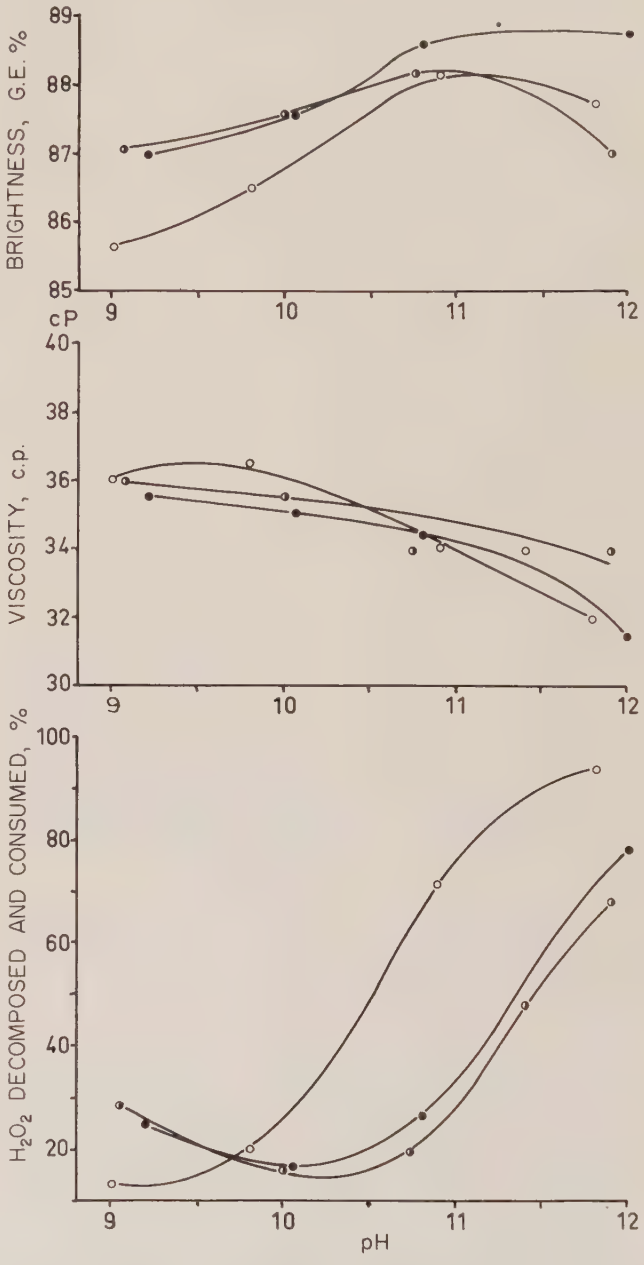

Fig. 10. Brightness, viscosity, and peroxide decomposition and consumption as a function of pH

○ 1% sodium silicate; ● 3% sodium silicate; ◑ 5% sodium silicate. H_2O_2 added, 0.3%; pulp consistency, 6%; distilled water; 4 hr; 70°C. Brightness of the pulp before the H_2O_2 stage, 81.6% G.E.; viscosity, 37 cp.

the reason why the brightness drops if the pH is increased above 11. There is also a sharp, undesirable drop in viscosity when the pH is as high as this.

Separate bleaching experiments were made at a very low pulp consistency, 0.5%, using the same concentration of HO_2^- ions at pH 11 and at pH 12. This was achieved by the addition of small amounts of hydrogen peroxide at intervals during the bleaching using the assumption that the degree of dissociation is 69% at

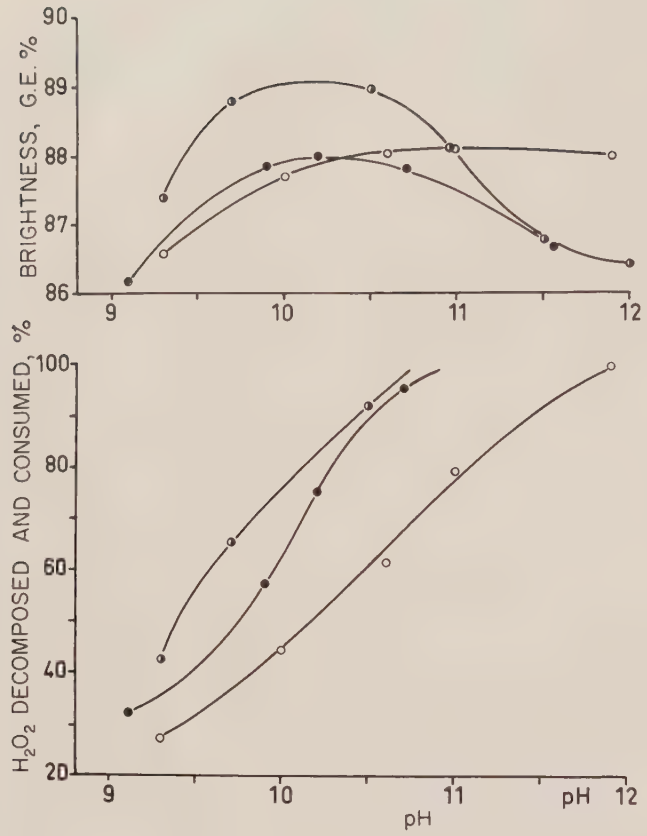

Fig. 11. Brightness, and peroxide decomposition and consumption as a function of the pH

○ 6% pulp consistency; 0.05% EDTA. ● 10% pulp consistency; 0.03% EDTA. ◑ 14% pulp consistency; 0.02% EDTA. H_2O_2 added, 0.3%; distilled water; 4 hr.; 70°C. Brightness of the pulp before the H_2O_2 stage, 79.0% G.E.

pH 11 and 93% at pH 12. As is shown in Fig. 9 the brightness obtained under these conditions was the same for both pH 11 and pH 12.

The Effect of Various Stabilizers

The addition of 0.05% EDTA or 0.05% magnesium sulfate did not affect the bleach consumption, the brightness, or the viscosity. In the present case, distilled water, analytical grade reagents, and polyethylene bottles were used and this is probably the reason why no effects were observed on addition of EDTA or magnesium sulfate.

The addition of sodium silicate with a $Na_2O:SiO_2$ ratio of 1:3.25 lessened the drop in viscosity even at a concentration of 1%, as is seen in Fig. 10, but 3% or more had to be added to obtain any appreciable decrease in bleach consumption. There was, however, no significant change in brightness when sodium silicate was added.

The effects observed may not be strictly typical for mill conditions since extremely pure water and reagents were used in our laboratory conditions.

Effect of Bleaching Temperatures

The effect of different bleaching temperatures: 60, 70, and 80°C., was studied using 3% sodium silicate as stabilizer in one case and 0.05% EDTA in another. In both cases there was an increase in brightness and bleach consumption and a decrease in viscosity with increase in temperature. The effect was much more pronounced when the temperature was increased from 60 to 70°C. than for the increase from 70 to 80°C.

Effect of Pulp Consistency

Three different pulp consistencies were used: 6, 10, and 14%. The results of these bleaching experiments using added EDTA are shown in Fig. 11. An increase in pulp consistency with a constant added amount of

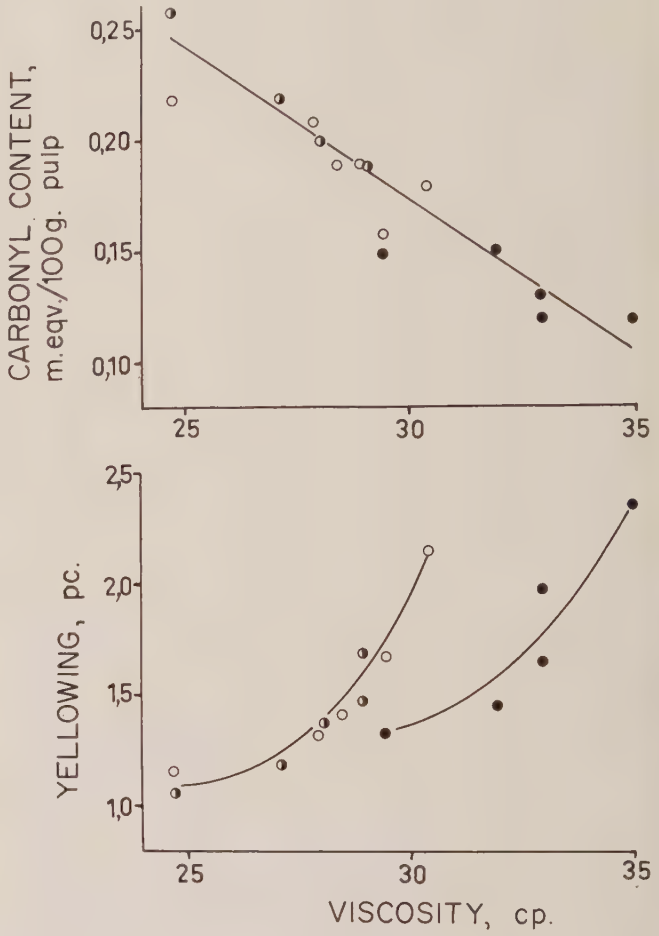

Fig. 12. Carbonyl content and yellowing as a function of the viscosity of the pulp after peroxide bleaching

○ H_2O_2 added 0.3%; ● H_2O_2 added 0.6%; ◑ H_2O_2 added 0.9%. 0.05% EDTA; distilled water; pulp consistency, 6%; 4 hr.; Brightness of the pulp before the H_2O_2 stage, 79.0% G.E.; viscosity, 37 cp.; carbonyl content, 0.12 millequiv./100 g.; carboxyl, 1.9 millequiv./100 g. pulp; *pc*-value, 3.59. Brightness of the pulp, 86.6–90.2% G.E.

bleaching agents means an increased concentration of the bleaching agent in the liquor and, therefore, a more rapid bleach consumption and also a higher brightness in the same bleaching time. At the highest consistency there was already a drop in brightness when the pH was increased above 10.5, probably because of insufficient residual hydrogen peroxide, as is shown in the lower diagram. A high consistency should consequently be favorable provided that the bleaching conditions are adjusted so as not to have too little residual hydrogen peroxide at the end of the bleach.

A study was also made of the effect of the amount of bleach added and of the bleaching time. An increased brightness was obtained with an increase in the amount of bleaching agent added but simultaneously there was a fairly marked drop in viscosity which in combination with the observed brightness increase seemed to in-

the present case was approximately 0.3%.

In all cases when the bleaching time was prolonged there was an increase in brightness provided that the final residual hydrogen peroxide was not too low. Otherwise there was a drop in brightness when the bleaching time was prolonged.

The Carbonyl and Carboxyl Content and Yellowing of the Pulp

The carbonyl content of the pulp was determined by the Geiger and Wissler method (*3*) with small modifications recommended by Wennerblom. The carboxyl

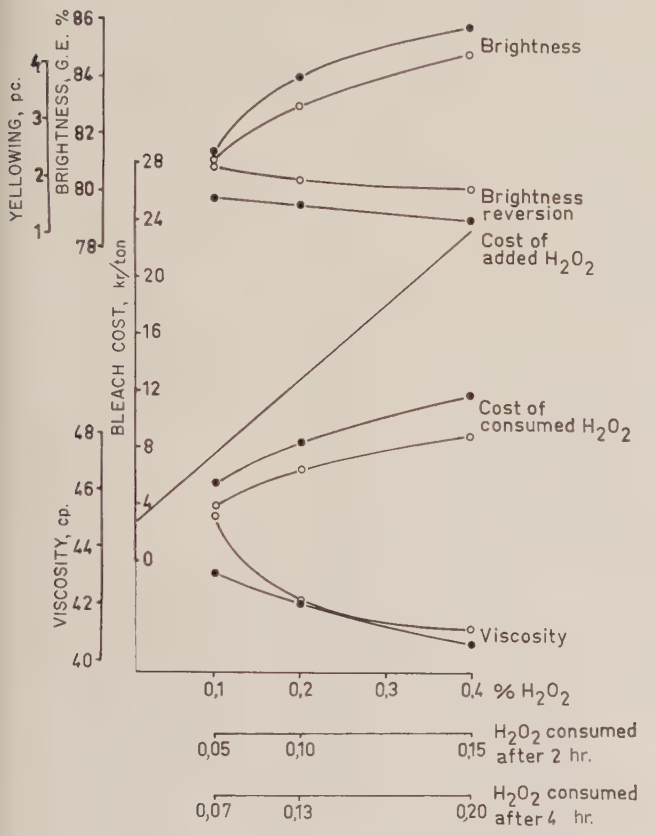

Fig. 13. Brightness, viscosity, pc-value, and bleach cost per ton of dry pulp as a function of the amount of H_2O_2 added (1 Kronor ≈ 19 cents)

Sodium silicate 0.35%; pH 10.0–10.5; distilled water; pulp consistency, 6%; 70°C. Brightness of the pulp before the final bleach, 78.8% G.E.; viscosity, 45 cp. ○ 2-hr. bleaching. ● 4-hr. bleaching.

content was determined by a dynamic method using an exchange of calcium ions with calcium acetate solution (*4*). The yellowing of the pulp was determined after heating at 120°C. for 16 hr. and the result was expressed as the *pc* number (*5*). As is shown in Fig. 12 the carbonyl content of the pulp increases during bleaching. However, as the viscosity decreases new carbonyl end groups are formed. Calculation showed that the amount of carbonyl end groups produced as the viscosity decreased was approximately equal to the observed increase in carbonyl content. The carbonyl groups produced should consequently not be situated at carbon atoms 2, 3, or 6 and, therefore, should not contribute to yellowing. As can be seen in the lower part of the figure the yellowing, on the contrary, decreases during bleaching and this result certainly cannot be explained by carbonyl groups. Some other factor must be the cause of the decrease in yellowing.

Determinations of the carboxyl content showed that it was approximately constant during the hydrogen peroxide bleaching and, thus, this also did not provide any explanations for the decrease in yellowing.

Comparison of Peroxide and Chlorine Dioxide Bleaching

The prebleached sulfate pulp used for this investigation was from a different batch from that used for the investigations described above. This pulp had a

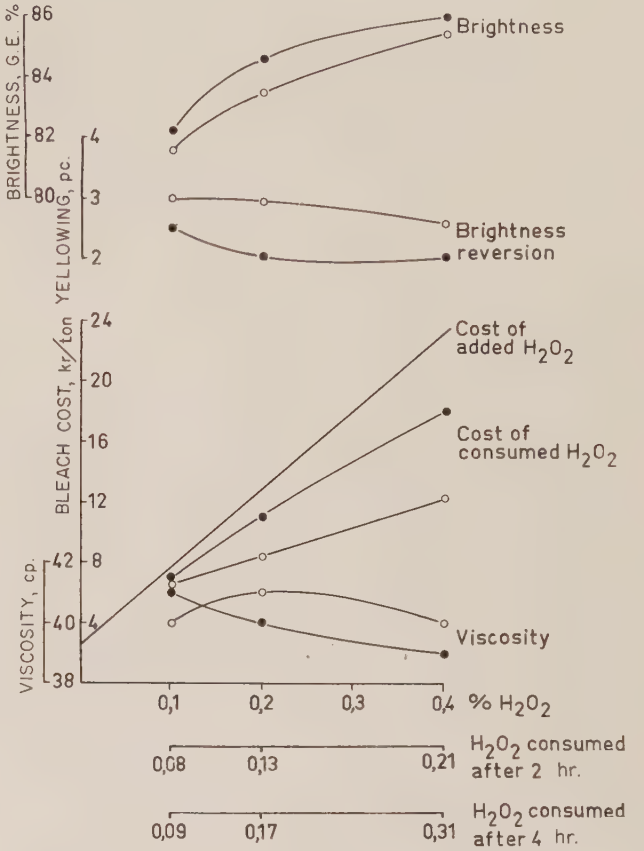

Fig. 14. Brightness, viscosity, pc-value and bleach cost per ton of dry pulp as a function of the amount of H_2O_2 added (1 Kronor ≈ 19 cents)

Sodium silicate 0.35%; pH 10.0–10.5; distilled water; pulp consistency, 14%; 70°C. Brightness of the pulp before the final bleach, 78.8% G.E.; viscosity, 45 cp. ○ 2-hr. bleaching; ● 4-hr. bleaching.

viscosity of 45 cp. TAPPI and a brightness of 78.8% G.E. Bleaching with hydrogen peroxide was performed under the following conditions: pH 10.0 to 10.5; temperature 70°C.; pulp consistencies, 6 and 14%; bleaching times, 2 and 4 hr.; peroxide charges, 0.1, 0.2, and 0.4%; distilled water with 0.35% sodium silicate. Bleaching with chlorine dioxide was done at: pH 4.7; temperature 70°C.; pulp consistencies, 6 and 14%; bleaching times, 2 and 4 hr.; and bleach charge, 0.2, 0.5, and 1.0% calculated as active chlorine. An economic calculation was made assuming the following chemical costs:

Peroxide (35% by weight)	35 cent/kg.
Chlorine dioxide (active chlorine)	15 cent/kg.
Sodium hydroxide	8.7 cent/kg.
Sodium silicate (aurosil N 31, 35% by weight)	3.2 cent/kg.

The results are presented in Figs. 13–16. Figure 13 shows the result of hydrogen peroxide bleaching at 6% consistency, giving brightness, brightness reversion, cost based on hydrogen peroxide added, cost based on peroxide consumed, and viscosity. Figure 14 gives the results for bleaching with hydrogen peroxide at 14% consistency and Figs. 15 and 16 show the results obtained with chlorine dioxide at 6 and 14% consistency, respectively.

A comparison of the peroxide and chlorine dioxide results shows that at 14% consistency a brightness of 86% G.E. was obtained with a charge of 1.0% chlorine

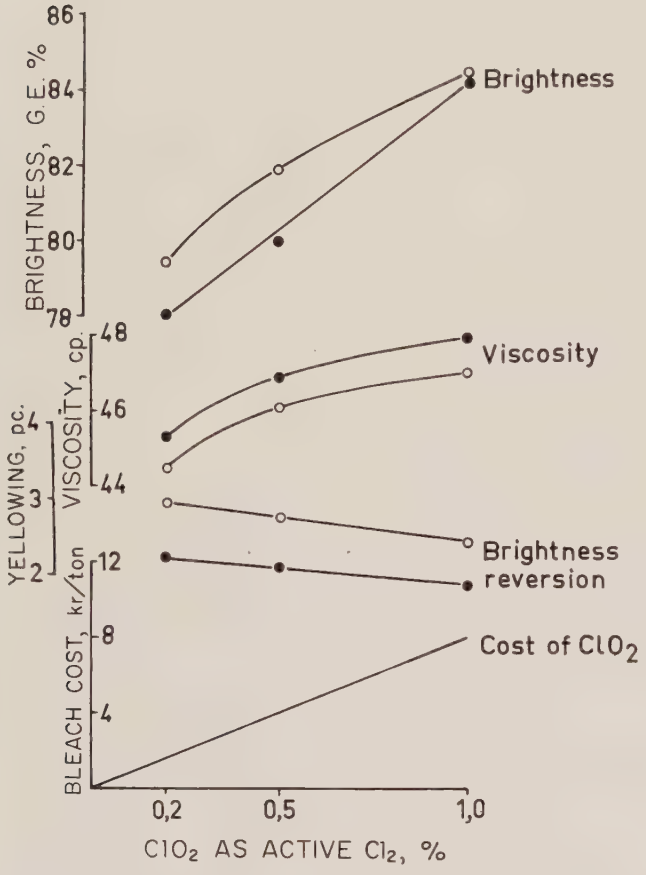

Fig. 15. Brightness, viscosity, pc-value, and bleach cost per ton of dry pulp as a function of the chlorine dioxide added, calculated as active chlorine (1 Kronor ≈ 19 cents)

pH 4.7; distilled water; pulp consistency 6%; 70°C. Brightness of the pulp before the final bleach, 78.8% G.E.; viscosity, 45 cp. ○ 2-hr. bleaching; ● 4-hr. bleaching.

dioxide calculated as active chlorine and 0.4% peroxide. At the same time the viscosity dropped by approximately 5 cp. units for the peroxide bleach but increased by approximately 7 units in the chlorine dioxide bleach. The brightness reversion was distinctly less for the pulp bleached with hydrogen peroxide than for the pulp bleached with chlorine dioxide.

The economic comparison shows, however, that bleaching with peroxide is definitely more expensive than bleaching with chlorine dioxide for the price levels given above. The price difference will be a little more favorable for the hydrogen peroxide if the cost is based not on chemicals added but on chemicals consumed. However, as long as the method of recycling and thereby re-using residual hydrogen peroxide is somewhat questionable the price calculations have to be based on the amounts of chemicals added.

CONCLUSIONS

In the bleaching of pulp with peroxide the maximum increase in brightness is usually obtained at a pH of approximately 11. A high brightness can also be obtained at a higher pH provided there is a certain minimum amount of residual hydrogen peroxide present. At the highest pH values, however, the decomposition of the hydrogen peroxide is so fast that after a certain

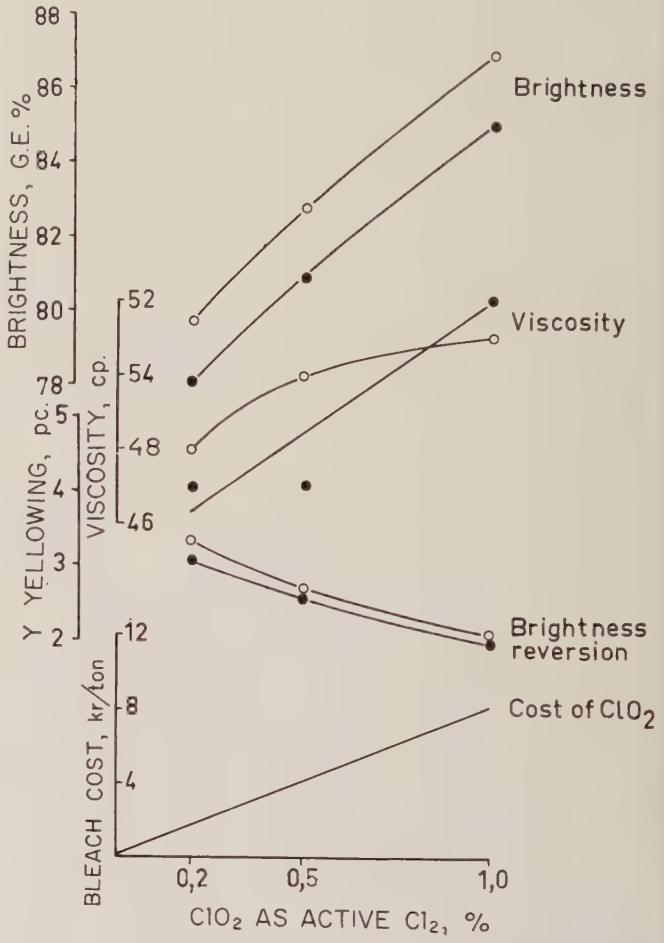

Fig. 16. Brightness, viscosity, pc-value and bleach cost per ton of dry pulp as a function of the chlorine dioxide added, calculated as active chlorine (1 Kronor ≈ 19 cents)

pH 4.7; distilled water; pulp consistency 14%; 70°C. Brightness of the pulp before the final bleach, 78.8% G.E.; viscosity, 45 cp. ○ 2-hr. bleaching; ● 4-hr. bleaching.

bleaching time there will usually be no appreciable residual hydrogen peroxide.

An increase in bleaching time is favorable provided there is some residual hydrogen peroxide present during the whole bleaching period. It is possible to obtain increased brightness with an increase in the amount of added hydrogen peroxide but the brightness increase gained by the additional hydrogen peroxide will be the smaller the greater the total amount of added hydrogen peroxide. In the present case there seems to be some sort of practical optimum at a charge of 0.3% hydrogen peroxide. The addition of sodium silicate decreases the amount of hydrogen peroxide consumed and in this case there would be a very favorable effect on the economics of the process if it were possible to

recycle the liquor from the hydrogen peroxide stage.

A high pulp consistency and a high temperature are favorable for bleaching.

Compared with chlorine dioxide bleaching hydrogen peroxide bleaching, for the same brightness, costs much more and gives a greater reduction in viscosity though the investment costs is less and a lower color reversion is obtained. It is a question of price versus quality, whether hydrogen peroxide or chlorine dioxide is to be preferred for the bleaching of kraft pulps.

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Characterization of Filler and Coating Pigments in Paper by X-Ray Techniques

I. Identification

CARROLL L. GAREY and JOHN W. SWANSON

X-ray diffraction patterns of handsheets containing low concentrations of kaolin clay, talc, TiO_2, and bentonite were prepared. The identification of the pigments was found to be clear-cut and the procedure easily standardized. Interference from the cellulose background was not serious except in the few cases where the major diffraction angles of the pigments occurred at or near the angles corresponding to the specified characteristic crystal spacings of cellulose. Diffraction patterns were also secured on sheets coated with coating colors containing kaolin clay, TiO_2, $CaSO_4$, $BaSO_4$, talc, and bentonite. The applicability of the technique is excellent when a pigment is present either alone or in mixtures. Diffraction patterns of the pigments determined from the coated sheets are more clearly defined than from filled sheets, due to the presence of a greater amount of the material per unit area in the beam for the coating than for the filled sheet. Preferential crystal orientation also contributes to the definition of the pattern in the coatings. A number of commercially available papers were examined by the diffraction method, and the data readily revealed the types of pigments used in their manufacture.

INTRODUCTION

The use of pigments in the filling and in the coating of many grades of paper and board is a common practice in the paper industry. As a result, problems are frequently encountered that require the identification of the materials present. Such information may have any of several purposes and may, if used properly, supply information that is related to the sheet composition variation, two-sidedness of the paper, orientation of the pigments, etc., of paper and board products.

There are a number of methods and procedures available for the identification of the fibers in paper (*1*, *2*). Methods for the identification of the various fillers (*3*) that may have been added to the pulp prior to making the sheet and of the coating materials (*4*) have been in use for many years. However, these and other methods in nearly all cases require the destruction of the sheet in order to analyze either chemically or physically for the various components. When samples available are small, this greatly limits the number and types of analyses that can be obtained from the given sample. Other methods for such identification and study would be of considerable value.

The use of the x-ray diffraction technique for the detection and identification qualitatively, and possibly quantitatively, of materials used in papermaking holds much promise. Other methods of examination are tedious and frequently are of the visual type requiring a microscope for observation. From a Utopian standpoint it would be ideal to be able to place a sample in a machine, push a button, and have the machine grind out an answer. X-ray analysis is deceivingly simple in that regard.

In 1912, Laue and his associates discovered that crystals act as diffraction gratings to x-rays. During World War I, two independent, widely separated laboratories discovered that powders of fine-grained crystalline materials gave characteristic diffraction effects (*5*). Much work in the scientific world on crystal structure has resulted and many characterizations have been made.

It is a well-known chemical phenomenon that the atoms in a molecule tend to arrange themselves in a definite manner that is a characteristic of the specific atoms involved. This definiteness of position, when repeated in adjacent molecules, tends to promote regularity of chemical arrangement resulting in structures that we call crystals. Any regularly, minutely spaced planes (or more correctly, atoms) in a chemical structure are capable of diffracting an x-ray beam incident upon them. At certain specific angles, the distances between these planes can be determined in accordance with Bragg's law (*5*)

$$n\lambda = 2d \sin \theta$$

where n is a whole number, λ is the wavelength of the x-rays, d is the distance between the diffracting planes, and θ is the critical angle of reflection at which the diffracting beam is in phase due to the whole-number multiple n of the wavelength.

Since crystalline materials have definite atomic spacings, the x-rays are reflected at definite angles and

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Many thousands of substances have been studied and the characteristic crystallographic spacings for many of them have been recorded in the ASTM Alphabetical Index for X-ray Diffraction Patterns (*6*) and in a number of books on the subject. Thus, when planar reflections appear on an x-ray pattern at certain specific angles, the presence of the material indicated by the spectrum of characteristic lines may be confirmed.

The rapid development of diffractometers (parafocusing geometry) utilizing the reflectance technique with Geiger counter detection since World War II has made this procedure useful in many areas (*5*). Recent advances in instrumentation have made application of the x-ray diffraction analysis method to powders straightforward and rapid.

Many materials are used by the paper industry for filler and for coating pigments. Nearly all of these are crystalline or have a regularly spaced molecular structure which will produce a diffraction pattern when irradiated with a beam of x-rays. The x-ray diffraction technique adapts itself to the crystalline materials used in papermaking and, thus, is a powerful tool to use in this field.

EXPERIMENTAL

Papers were prepared in the laboratory to provide samples of both filled and coated material for examination by x-ray diffraction technique. A Weyerhaeuser bleached sulfite pulp was used to form handsheets filled with various types of filler materials. A 50% groundwood raw stock containing no filler was used as a coating base stock.

Handsheets were prepared in the laboratory which included several types of filler pigments. The pulp slurry was beaten at 1.5% concentration in a Valley beater to a Schopper-Riegler freeness of 500 cc. The slurry was then diluted to 0.5%, sized with 3% pale rosin size and then 4% alum, and stirred for 5 min.

Two per cent of filler was added based on the pulp present and the slurry mixed for 30 min. A Noble & Wood handsheet mold was used to form sheets, the water in the headbox having been adjusted to pH 4.5. Sheets were made to a basis weight of 45 lb. for a 25 × 40–500 ream, pressed at 50 lb. per sq. in. for 5 min. and drum-dried on a steam-heated drier for 7 min. at $3^1/_2$ p.s.i. of steam. Specific filler retention was not determined.

The coated paper samples were prepared by first making dispersed slips of the pigments, and adding 17 g. of hypochlorite-oxidized starch per 100 g. of the pigment as the adhesive. All pigments with the exception of TiO_2 and $CaCO_3$ were dispersed at 72% solids using 3 meq. of Quadrafos and 2 meq. of NaOH per 100 g. of pigment. The $CaCO_3$ and TiO_2 were dispersed with 1% Calgon-T. Pluronic F-68 was used as a wetting agent for talc. The starch was precooked at 25% solids at 90 to 95°C. for 15 min. and cooled before mixing into the color. The final colors were diluted to 50% total solids and applied to the wire side of the raw stock with a Mayer rod. Coat weights were held to 18 lb. per 25 × 38–500 ream.

Diffraction patterns were obtained with a North American Philips x-ray unit (Philips Electronics, Inc.), the samples being scanned with the Norelco with a Brown recording potentiometer. Radiation (K_α) from a copper target tube, operated at 35 kv. and 20 ma. with a nickel filter, was used throughout this work. The x-ray beam was collimated with $^1/_4$° divergence and scatter slits and a 0.006-in. receiving slit. The scale factor, multiplier, and time constant of the rate meter in the counting circuit were set at 1, 1, and 16, respectively, for samples containing pigment filler and at 2, 0.6, and 16, respectively, for coated samples in order to obtain good resolution of the diffraction peaks relative to the background. In usual practice, these factors may be adjusted as required for the individual samples. Scans of the diffraction spectra were made from an angle of 4°2θ to approximately 50°2θ in order to obtain a coverage of the important diffraction lines for most materials to be encountered.

Measurements of the reflected angles were made, the crystalline d spacing calculated according to Bragg's law, (conversion tables are available), and the relative line intensities noted. Examination for resolution of line patterns above background and between closely spaced lines was made.

Table I. Diffraction Angles and Corresponding *d* Spacings of Selected Filler and Coating Pigment Materials Used in Paper (Cu X-ray Target—K_α Radiation)

Pigment	*Bragg angles, 2θ°*	*Calcd. d spacing, A*
Cellulose	12.5–19.7	7.0–4.5
	22.8	3.9
Clay, kaolin	12.35	7.15
	19.95	4.45
	24.95	3.57
	37.9	2.37
Talc	9.8	9.00
	19.3	4.60
	28.6	3.12
TiO_2, anatase	25.4	3.51
	37.8	2.38
	48.2	1.89
TiO_2, rutile	27.5	3.24
	36.1	2.49
	54.2	1.69
Bentonite	6.0	14.7
	7.3	12.2
$2\,CaSO_2 \cdot H_2O$	14.5	6.10
	29.5	3.03
	31.7	2.82
$CaSO_4 \cdot 2H_2O$	11.7	7.56
	20.8	4.27
	29.2	3.06
$BaSO_4$	25.9	3.44
	26.8	3.32
	28.8	3.10
	42.7	2.12
Paraffin	11.8	7.50
	19.3	4.60
	21.5	4.13
	23.7	3.75
	29.5	3.03
	38.8	2.32

Unknown samples were examined by the above described x-ray diffraction technique and the identifications of the materials present were made with the aid of the ASTM Index (*6*) or other known record. In most cases, the three most intense lines are satisfactory for identification and can be further confirmed with the remaining lines from the index cards.

The most characteristic diffraction lines of the materials used in these investigations are given in Table I.

RESULTS

The identification and study of the various components of paper has been shown to lend itself quite well to x-ray diffraction techniques. Data are obtained from a measurement of the angles of reflection and also from line intensities of the reflected beam. The intensity of the signal from the diffracting plane is related to the number of planes in a position to cause a given reflection. Thus, in perfectly random samples, the intensity theoretically should be proportional to the amount present. There are a number of sample and condition variables that affect the intensity (*5*) and, therefore, a quantitative interpretation of diffraction must be made with considerable reservation. These are usually 'best estimates' based on standards, sample preparation methods, and experience.

Cellulose fibers have a varying degree of crystallinity depending upon their source and upon their processing history (*7*, *8*). However, the diffraction pattern of the basic cellulose crystallite is easily distinguished by its planar spacing calculated from the diffraction angles and by the shape of the diffraction bands. A diffractogram from a handsheet made of Weyerhaeuser bleached sulfite and containing no filler or coating materials is shown in Fig. 1. Cellulose-I produces a

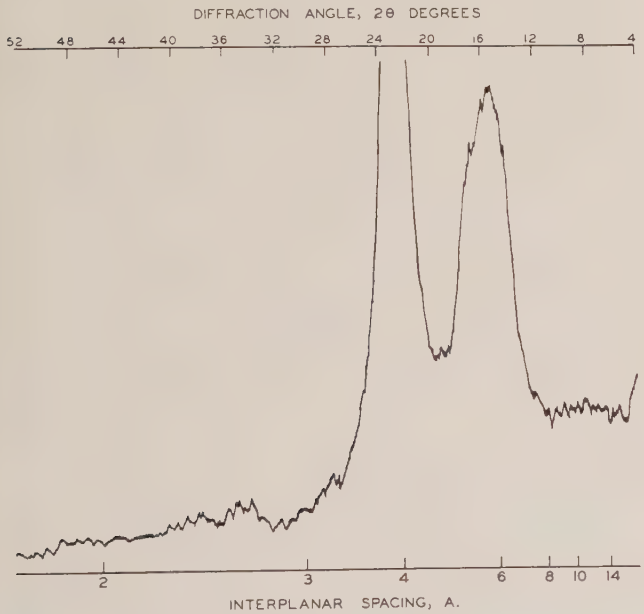

Fig. 1. Radial x-ray diffraction trace of cellulose-I as a laboratory handsheet. (Cu Kα radiation)

broad double peak [Miller Indices of (101) and (10$\bar{1}$)] in the region from 7.0 to 4.5 A. and a sharper, more clearly defined peak at approximately 3.9 A. (002). Cellulose absorbs x-rays only weakly, so that little interference is encountered for the determination of diffraction patterns of filler materials, even when they are present in small proportions. The pattern for cellulosic material is quite distinctive and it is rarely confused with any other materials.

Filled Papers

The use of most pigment filler materials will produce an additional diffraction pattern from x-rays and such a pattern can be seen in the presence of cellulose.

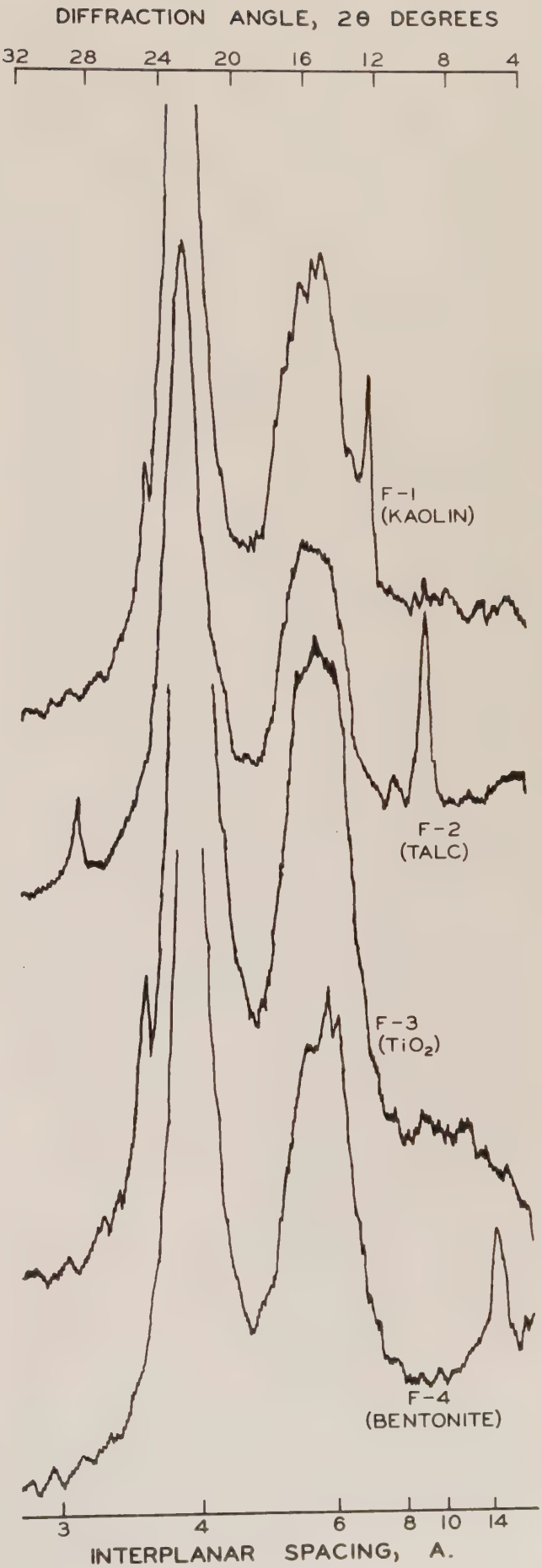

Fig. 2. Radial x-ray diffraction trace of cellulose handsheets containing several types of filler materials. (Cu Kα radiation)

Diffraction patterns are reproduced in Fig. 2 for handsheets containing low concentrations (2%) of several pigments. Kaolin clay as illustrated by trace F-1 has a definite pattern easily identifiable at planar spacings of 7.2 and 3.6 A., and even at this low concentration is clearly defined and specific for this material. There are some additional peaks that would normally show at 4.5 A. but those weak lines are hidden by the cellulose pattern. Additional lines would show at higher angles (smaller d spacings) if the scan of the diffractometer were to be extended.

The presence of talc in a cellulose sheet produces a diffraction pattern that is specific for planar spacings at 9.0 to 9.4 A. and at 3.12 A. There are several forms of magnesium silicates that could be considered but the most common forms that are found in paper have the spacings indicated above, plus one additional peak barely seen in Fig. 2, F-2 at 4.6 A. (19.3°2θ). However, the first two peaks mentioned have the highest intensities and are located at positions at which there is no cellulose interference.

The identification of the anatase form of TiO_2 in trace F-3 is made possible by the characteristic lines at 3.51 A and at 2.39 and 1.89 A. not shown. Anatase is easily distinguished from the rutile form which has lines occurring at 3.24, 2.49, and 1.69 A. Some confusion of the largest TiO_2 (anatase) spacing with the (002) spacing of kaolin (3.6 A.) would be expected since they occur close together. However, both lines are strong and since these are crystalline materials of usually good quality, the lines are sharp and independent as will be seen later.

Bentonite, in small amounts, is often added to certain types of papers and, as shown in curve F-4, is identifiable by its planar (001) spacing at 14.0 A. in airdry condition. Since this is an expanding type mineral, the amount of moisture present in the sample will determine the exact line spacing observed. The ovendry bentonite spacing may be as low as 12 A. and in a moist or wet sample, the line will either move to larger d spacings or become diffuse as not to be seen. Thus, this may serve as an additional test for the presence of this material. The identification and estimates of bentonite are tricky, but it can be identified

Table II. Pigment Composition of Laboratory Coatings Used to Study X-ray Diffraction

Coating no.	*Type of pigment*	*Composition, %*
C-1	Kaolin clay	60
	TiO_2, anatase	20
	$2CaSO_4 \cdot H_2O$	20
C-2	Kaolin clay	75
	$CaSO_4 \cdot 2H_2O$ / $2CaSO_4 \cdot H_2O$	25
C-3	Kaolin clay	75
	$BaSO_4$	25
C-4	Kaolin clay	73
	TiO_2, anatase	10
	$2CaSO_4 \cdot H_2O$	10
	Talc	5
	Bentonite	2

in the presence of cellulose at concentrations of 0.25% or less, based on cellulose. If kaolin clay is also present, the weak reflection of small amounts of bentonite will be absorbed and it usually cannot be identified in amounts less than 2%, based on the clay present.

Coated Papers

Diffraction patterns were also secured on papers coated with colors containing various pigments, Fig. 3. The application of x-ray identification is excellent when a pigment is present both alone and in mixtures. Diffraction patterns of the pigments are more clearly defined from coated sheets than those from filled sheets. This is due, in part, to the presence of a greater amount of the material per unit area in the beam for the coating

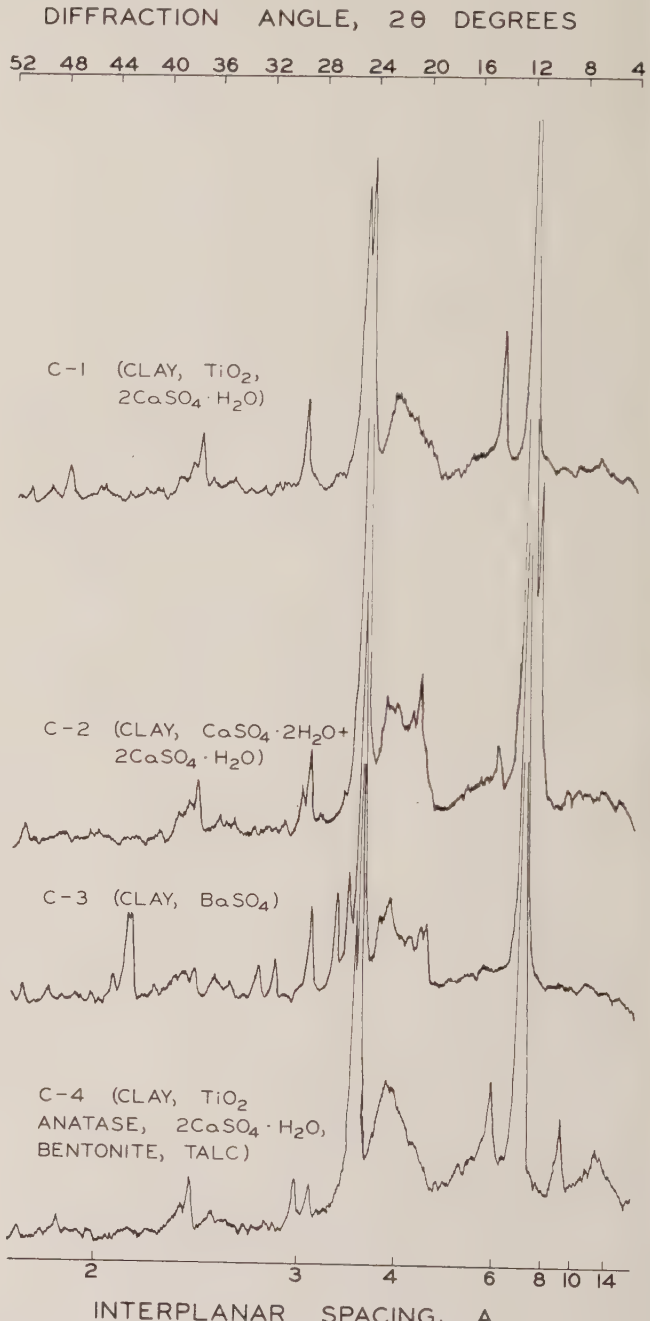
DIFFRACTION ANGLE, 2θ DEGREES
52 48 44 40 36 32 28 24 20 16 12 8 4
C-1 (CLAY, TiO2, 2CaSO4 · H2O)
C-2 (CLAY, CaSO4 · 2H2O + 2CaSO4 · H2O)
C-3 (CLAY, BaSO4)
C-4 (CLAY, TiO2 ANATASE, 2CaSO4 · H2O, BENTONITE, TALC)
2 3 4 6 8 10 14
INTERPLANAR SPACING, A.

Fig. 3. Radial x-ray diffraction trace of laboratory-coated paper using a variety of pigments. (CuK$_\alpha$ radiation)

than for the filled sheet. Also, since the coating is on the surface, reflection from the cellulose base sheet is minimized due to absorption of the diffracted beam by the pigments. It is known that preferential crystal orientation in the coating, such as in the case of plate-like pigments, may also contribute to definition of certain lines in the pattern.

All of the traces reproduced in Fig. 3 for coated sheets contain kaolin clay as the basic pigment. Other

materials were included in coating colors as indicated in Table II. It should be noted that the cellulose pattern is almost completely masked since a coat weight of 18 lb. per 25 × 38–500 ream was purposely used to hide the background. Small remnants of the peak at 3.9 A for cellulose can be seen on all four diagrams.

Figure 3, C-1 is a diffraction diagram of a coating containing clay, TiO_2, and $CaSO_4$ in proportions by weight of 60:20:20. The characteristic clay lines are readily identifiable by the spacings found at 7.2, 3.6, and 2.37 A which are the three most intense peaks for this material. The TiO_2 is recognized by lines at spacings of 3.51, 2.38, and 1.89 A which are those for the anatase form of this material. It is apparent that while observed peaks for clay and for TiO_2 at 3.6 and 3.51 are very close, they are readily resolved by the diffraction detection system. The $CaSO_4$ lines are of interest. These occur at 6.10, 3.03, and 2.82 A and are specific for that particular form having a formula of $2CaSO_4 \cdot H_2O$.

In order to illustrate the identification of different hydrated sulfates, a coating was prepared using clay and a technical grade of $CaSO_4$ instead of a c.p. grade. This diffractogram is shown as C-2. In addition to the lines for kaolin clay, lines for two forms of $CaSO_4$ are readily identified. These forms are (1) $2CaSO_4 \cdot H_2O$ at spacings of 6.10, 3.03, and 2.82 A. as seen previously, and (2) $CaSO_4 \cdot 2H_2O$ at spacings of 7.56, 4.27, and 3.06 A. Clearly, the interpretation depends upon an understanding of the crystallographic structures and a previous knowledge of spacings for each type. Such data are readily available in the ASTM Index (*6*).

Curve C-3 in Fig. 3 is a trace of a coated paper containing kaolin clay and $BaSO_4$ in a ratio of 75:25. The clay lines are clearly defined. The line pattern for $BaSO_4$ is unique for a coating pigment. In this case, several lines are close together and are sharp and distinctive so that a definite identification can be made where they appear at 3.44, 3.32, 3.10, and 2.12 A. These lines are also well defined when examination is made of many photographic papers in which $BaSO_4$ is a common component. Silver compounds may also be identified and located in such paper sheets by this technique.

In order to see just how complex the coating color components could be and still obtain clear diffraction diagrams, a coating was prepared that gave the diffraction pattern in C-4. Kaolin clay dominates the pattern but TiO_2 may be independently observed at 3.51 A as a side peak on the (002) kaolin line (3.6 A.). The $2CaSO_4 \cdot H_2O$ form is clearly defined at 6.10 A. as is talc at 9.4 A. Bentonite was included in this mixture and shows at about 14 A. but the line is broad in this region. This line is not as clear-cut as would be desired. Identification could be further confirmed by solvating the system with water to expand the lattice of this mineral to higher spacing or with glycerol to cause the line to move to 17.7 A. The proportions of the materials present in this coating are, in the order named, 73, 10, 10, 5, and 2%.

Special Samples

A number of papers obtained from random sources on the commercial market were examined for their identifiable crystalline components. Sample S-1 in Fig. 4 is a 50% rag bond paper containing filler materials of talc and TiO_2. No clay can be identified in this sample. There is a good pattern for cellulose since the filler materials are not present in a large enough quantity to completely hide the reflection.

Trace S-2 is for a bread wrapper. In this case care must be taken in the observations to avoid confusion. The cellulose pattern is obvious for the regions from

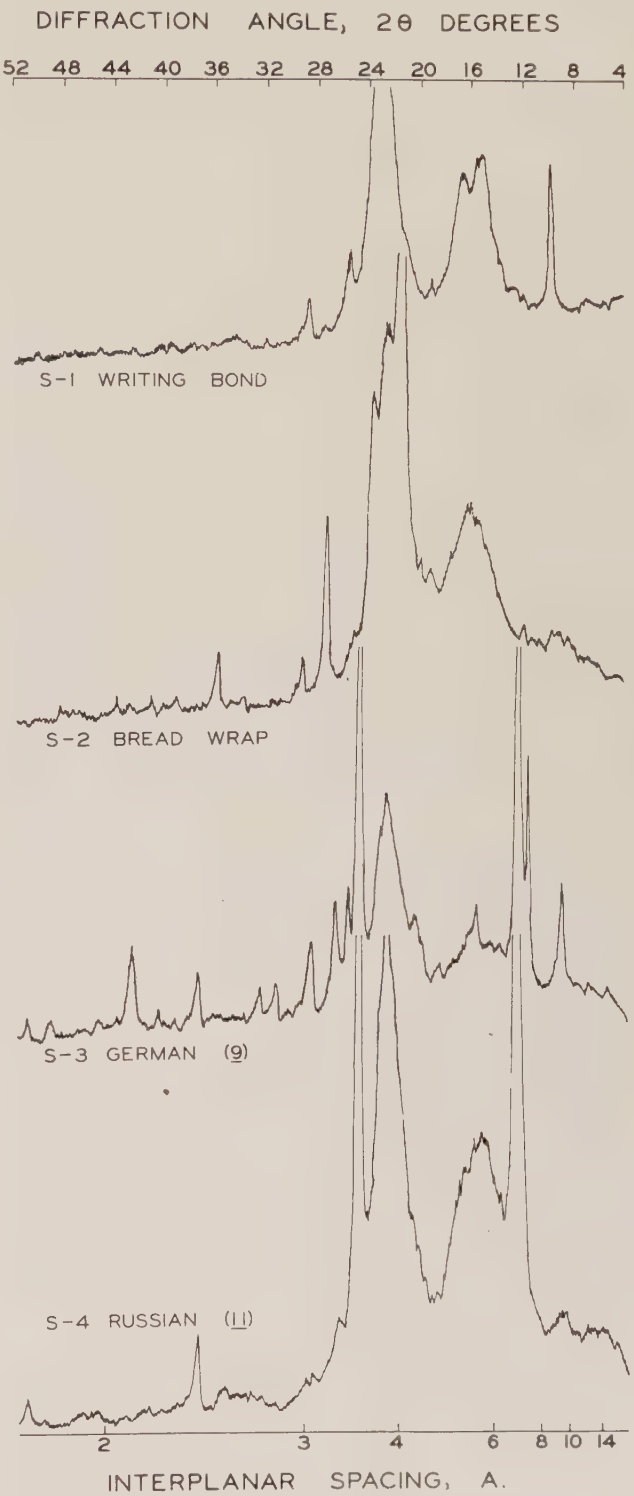
DIFFRACTION ANGLE, 2θ DEGREES
52 48 44 40 36 32 28 24 20 16 12 8 4
S-1 WRITING BOND
S-2 BREAD WRAP
S-3 GERMAN (9)
S-4 RUSSIAN (11)
2 3 4 6 8 10 14
INTERPLANAR SPACING, A.

Fig. 4. Radial x-ray diffraction trace of papers selected from several commercial sources for identification of filler and coating materials. (Cu K_α radiation)

7 to 4.5 A and 3.9 A. Superimposed upon this trace are the lines for TiO_2, but in this case the identification is for the rutile form at 3.24, 2.48, and a faint line at 1.75 A. Further lines may be observed such as the indefi-

nite peaks or lines seen at 7.50 and 4.60 A. Very strong lines at 4.13 and 3.75 A. combine to almost overshadow the 001 line for the cellulose at this point. Other lines at 3.03 and a small one at 2.32 confirm the identification of this material as paraffin which is known to have a crystalline structure and to give good diffraction lines. No clay was found in this sample.

As a matter of interest, two additional samples of commercial papers were examined. The first of these was a coated paper from a publication produced in Germany (*9*). The lines indicate the probable presence of several materials, a mixture of kaolin clay, talc, $BaSO_4$, and $CaSO_4 \cdot 2H_2O$ at the specific spacings as outlined previously (Table I). While it is true that many papers in Germany are coated with colors that include satin white as a pigment, this material has been shown to form an amorphous powder when dried at 105°C. (*10*). The diagram shown contains definite diffractions for kaolin that are not related to satin white. Possible complexes formed by the satin white precipitation conform to certain of the spacings for talc and $CaSO_4$ (*6*). The structure of satin white is not known (*10*) with certainty but it is believed that these traces show kaolin clay, satin white, and $BaSO_4$ in this paper.

Trace S-4 is of interest since it is for a paper sample from a Russian publication (*11*). This sample contains kaolin clay as the pigment and no other materials could be identified. The line intensities and dominant cellulose pattern indicate a paper filled with this material but the sheet was of low quality and brightness.

DISCUSSION

Examination of any paper sample requires a sufficient mass of the material in the beam to give a satisfactory reflection intensity. Ten thicknesses of the paper sheets with caliper of 3 mils have been found to be ideal in order to give reproducible diagrams. This will vary considerably with the basis weight. Any layers above two to three usually do not serve to greatly increase the intensity of reflection but rather they aid in resolution and avoid erratic intensities of the resultant diffractogram. It is, however, quite possible to use a single layer of the paper sheet and to determine the identity of crystalline materials present. Orientation and positioning of samples, especially in the case of small or single layer samples, is very important. This determines better area impingement of the beam and gives more satisfactory resolution of the peaks.

The geometry of a scanning goniometer of the type used in these experiments is of importance in the considerations of reflection intensity and instrument resolution.

With small odd-shaped specimens, the x-ray beam at low angles will completely bathe the sample and part of the beam will actually miss the sample. As the angle increases, the beam converges on the sample proper providing better resolution and intensity at the slightly higher angles than at the initial low angles. The actual intensity of irradiation per unit area increases sharply and, therefore, the reflected intensity increases with angle. However, further scanning converges the beam on a smaller sample area where there are fewer reflecting surfaces in the beam path. Therefore, the intensity of reflection as recorded decreases. Instrumentally, adjustments can be made in the goniometer by proper selection of slits to correct for these limitations.

Identification at low 2θ angles becomes extremely difficult due to resolution of the lines. Lines that would be seen at such angles as $6°2\theta$ are difficult to resolve, particularly on small specimens. This would also be true at $12.5°2\theta$ where the interference with cellulose background would be quite high. Thus, it has been observed that the relative peak heights and areas under these peaks are not consistent between samples of small size. In preliminary positioning examination, this was confirmed by changing slightly the position of the same sample in the goniometer. Therefore, repetition between the various paper samples using small clippings of the order of $1/8 \times 1/4$-in. rectangles and especially triangles and single layers of the sheet have been found to be slightly inconsistent. However, the positions of the various peaks will tend to show, regardless of sampling, if the materials are present.

One of the most distinctive advantages inherent in x-ray diffraction work, in addition to that of small sample requirement, is that the sample is not destroyed during analysis. This readily permits other analyses to be made on the same material. In addition, one of the most rewarding features of this type of analysis is that the man-hour time requirement is low for the information that can be obtained.

It should be pointed out that the simplicity of x-ray diffraction work is deceiving in actual usage. The interpretation of the diagrams for a component sample or many phased systems is quite complex. This may lead to erroneous interpretations unless other methods of analysis are used in conjunction with diffraction as guides.

Fortunately, the materials that may be expected to occur in paper as fillers and as coatings usually limit the possible interpretation of data. Certain diffraction patterns reflect the interference of diffraction among some materials as was pointed out in the case of kaolinite and bentonite. The cellulose pattern is large, broad, and irregular and care must be used to scan critical areas with sufficient sensitivity to obtain accurate instrument response. Several materials may have diffraction lines at or near equivalent values. Thus, care must be used to interpret these separations. It is important that as many lines as possible for each critical material be present in order to make any of the identifications positive.

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Brightness of High Yield Pulps

II. Reductive Bleaching of Cold Soda Pulp from White Birch with Sodium Borohydride

PHILIP LUNER

The ultimate brightness of a cold soda pulp from white birch (*Betula papyrifera* Marsh) with an initial brightness of 45% (Hunter) was found to be 74% using a large excess of sodium borohydride. From the rate of brightness development it appeared that either two types of reducible groups are present or that the chemical and/or physical accessibility of the pulps governed the rate of brightness development. The effect of chemical accessibility on the rate of brightness development was determined by experiments conducted in alkali of various concentrations and solvents of different swelling capacities. The effect of physical accessibility was studied by the variation in brightness development and chemical consumption at different freeness levels of the pulps. Investigation of the effect of concentration of chemicals, consistency of the pulps and reaction temperature, on the rate of brightness development indicates that high temperature bleaching under alkaline conditions gives the best utilization of chemical. Neutral salts were not effective as catalysts in the brightness development during sodium borohydride bleaching. Sodium borohydride is also effective in preventing the formation of colored bodies during the preparation of the cold soda pulp.

In recent years the trend in the paper industry has been towards the manufacturing of high yield pulps and new pulping methods have been found retaining larger amounts of carbohydrate material in the pulp. At the present time there is a great interest in exploring newer methods of pulp manufacturing which also retains part of the lignin. The two main problems encountered with pulps containing lignin are the lower brightness and strength. For many purposes the strength achieved with these mechanical type pulps are adequate. However, more extensive use of these pulps could be made if improved economical bleaching methods were available. The long-range aim of the present investigation is therefore to obtain a more basic understanding of the chromophoric groups responsible for the color of wood and wood pulp and the chemical changes incurred in these groups with bleaching chemicals.

CHROMOPHORIC GROUPS INVOLVED IN BLEACHING HIGH YIELD PULP

The chemicals most commonly used in bleaching pulps containing appreciable amounts of lignin are hypochlorite, peroxide, and hydrosulfite. These chemicals are used in single stage or multistage processes. The bleaching conditions used to derive their optimum benefit are described in the literature (*1*, *2*, *3*), and hence this phase of bleaching will not be reviewed here.

It is somewhat surprising, in view of the wide applicability and extensive experimentation with the hypochlorite, peroxide, and hydrosulfite systems, to note the lack of basic data regarding the chemical action of these chemicals on the noncarbohydrate portion of wood. This no doubt originates in part from the complex and partly unknown structure of lignin.

The reaction between hypochlorite and lignin has been studied by Richtzenhain, (*4*), Sacks (*5*), and Brounstein (*6*). The results showed that hypochlorite attacks lignin primarily through the free phenolic group. However, a limited amount of degradation can be obtained even if these groups are etherified when oxidizable side chains are present. This has been elaborated on in studies with lignin model compounds by Richtzenhain (*7*) and Sutherland (*8*).

The interaction of hypochlorite with wood is a topochemical reaction (*9*) and those wood components whose surface is exposed or close to the surface will react first. Since in high yield pulps lignin covers large areas of the outer fiber surfaces, lignin is attacked in preference to the carbohydrates in hypochlorite bleaching. The net result is an increase in brightness. Clearly, chromophoric groups are removed in this reaction.

A further indication of the chromophoric groups involved in bleaching was made when Brauns (*36*) found that methylation of unbleached kraft and sulfite pulp gave a 16 point brightness increment. In bleaching the methylated pulp with hypochlorite the reaction rate was retarded and less chlorine was consumed. Brauns suggested that the same hydroxyl group that is methylated is responsible both for the brightness increment and the retardation in the rate of hypochlorite bleaching. In view of the recent reactions involving diazomethane (*10*) and recent progress in lignin chemistry, it would seem that the brightness increment on treatment with diazomethane is due to the elimination of the quinone groups by the formation of pyrazoline groups in both the kraft and sulfite pulp, and that the retardation in the hypochlorite reaction is due to the etherification of the free phenolic groups (*9*).

Information on the structural changes occurring in peroxide bleaching was investigated by Jones (*11*). No great changes in the wood analysis were found due to the reaction with peroxide. The peroxide consumed was distributed as 40% lignin and 60% holocellulose. The extractives (ethanol and ether) accounted for only 1 to 4% of the total consumption. Since methylation with diazomethane inhibited the peroxide reaction with native lignin, it was deduced that peroxide attacks lignin primarily through the

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carbonyl group in the lignin resulting in a brightness increment (*11*).

The reactions occurring due to hydrosulfite treatment of wood or pulps with high lignin content have only been investigated in a qualitative manner (*12*). The reactions involved are largely unknown, but in view of the type of wood species used it would seem that part of the brightness increments results from the reduction of the highly colored extractives.

REACTIONS OF SODIUM BOROHYDRIDE AND WOOD COMPONENTS

Since sodium borohydride has become available it has been extensively employed in the field of cellulose chemistry where the role of carbonyl groups on brightness reversion, (*13*, *14*), acid, and alkali hydrolysis have been studied (*15*, *16*, *17*). The interaction of sodium borohydride with lignin has received much less attention. Smith (*18*) showed that the reduction of four native lignins caused a decrease in the extinction coefficient at 350 mμ. In each case a rapid stage, concluded in 30 min., was followed by a slower stage extending several hours. By comparison with model compounds the initial fast reaction was ascribed to 4-0-substituted cinnamaldehyde groups, and the slower reaction to parahydroxy-phenylketone groups. Adler and Marton (*19*) extended this work to milled wood lignin and again, in conjunction with the rates of reduction of additional model compounds, the number and type of carbonyl groups were determined. Gierer (*20*) determined the amount of carbonyl groups in native lignin by measuring the sodium borohydride consumption as was done for cellulose by Lindberg (*21*). These values found were twice as high as those discovered by Adler and Marton (*19*). In these investigations no mention was made of any relationship between the extent of reduction and the color of the lignin.

Donofrio and Mayer (*22*) were the first to observe that reduction of spruce groundwood with sodium borohydride resulted in a significant increase in brightness. The final brightness depended on the bleaching conditions but a 10 point brightness increment could be obtained with 2% chemical at 35°C. These authors suggested that the brightness increment was due to reduction of alpha-carbonyl groups in lignin. Reflectivity measurements showed that the sodium borohydride treatment gave higher reflectivity values in the 300 to 400 mμ region than peroxide or hydrosulfite.

From the foregoing survey it may be concluded that the chemical changes induced by hypochlorite and peroxide are largely unknown and complicated. Nevertheless, carbonyl groups have been shown to be involved in a number of bleaching reactions. Since sodium borohydride reduces only carbonyl groups it may lend itself better than other chemicals, whose action is nonspecific, in evaluating the relationship between chemical constitution, e.g., carbonyl groups and brightness.

EXPERIMENTAL

Procedure and Methods

White birch chips (*Betula papyrifera* Marsh) soaked in 50 g.p.l. aqueous sodium hydroxide for 1 hr., were placed in a mechanical kneader with additional caustic (50 g.p.l.) for 1 hr. The pulp was further disintegrated with a high speed mechanical stirrer to the desired freeness. This procedure to lower the freeness was used when a stainless steel disk refiner was not available. The pulp was then washed with distilled water to neutral pH and adjusted to pH 5 with hydrochloric acid. Bleaching with sodium borohydride at low consistency was done in beakers, at medium consistencies in polyethylene bags, and at higher consistencies the chemical was sprayed directly upon the wet pulp pads. The bleaches were done at the temperatures as indicated.

At the end of the bleaching time, where possible, a sample of the liquor was analyzed for residual sodium borohydride. The pulp was then washed with distilled water to neutral reaction and adjusted to pH 5 with hydrochloric acid before optical handsheets were made according to TAPPI Standard T 217 m-48. Brightness measurements were made on a Hunter Multipurpose Reflectometer using the blue tristimulus filter.

In the investigation yields of bleached pulps were not determined as there was no indication that lignin is removed by the sodium borohydride reduction. However, it is expected that some losses in yield have occurred due to carbohydrate losses in the bleaching experiments containing alkali.

Standardization of the sodium borohydride solutions and solution analysis were done as previously described (*23*). The copper number was determined according to the Swedish Standard CCA 3 (Hägglund modification) using 1-g.-samples.

RESULTS

Preliminary results indicated that large brightness increments could be obtained by bleaching cold soda pulp with sodium borohydride. It was consequently of interest to determine the ultimate brightness attainable of a cold soda pulp reduced with sodium borohydride.

The bleaching conditions employed were room temperature, 10% chemical based on ovendry pulps, and 10% consistency of the pulp slurry. Both alkaline (0.1 *N* NaOH) and unbuffered conditions were employed. These results are shown in Table I. It may be seen that the brightness of the buffered bleach increased continually while in the unbuffered bleach a small decrease in brightness is observed at the longer time intervals. This decrease in brightness was prevented and higher initial brightness values were obtained under unbuffered conditions if the cold soda pulp was pretreated at pH 3 with hydrochloric acid for 2 hr. Additional brightness increments can be

Table I. Ultimate Brightness Values of Cold Soda Pulps Reduced with Sodium Borohydride at 25°C. and 10% Consistency

	Conditions	*Time, hr.*	*Brightness—Hunter, %*							
			0	*4*	*6*	*6*	*23*	*96*	*65*	*90*
1.	0.1 *N* NaOH	...	45.0	56.5	59.3	60.7	63.3	66.8	..	..
2.	Unbuffered	...	40.0	63.0	63.6	65.6	61.0	61.4	..	..
3.	Unbuffered pretreated with HCl	Stage I	45.0	66.3	68.1	68.9	68.1	66.9	..	..
		Stage II	66.9	..	..	..	..	..	74.1	73.0

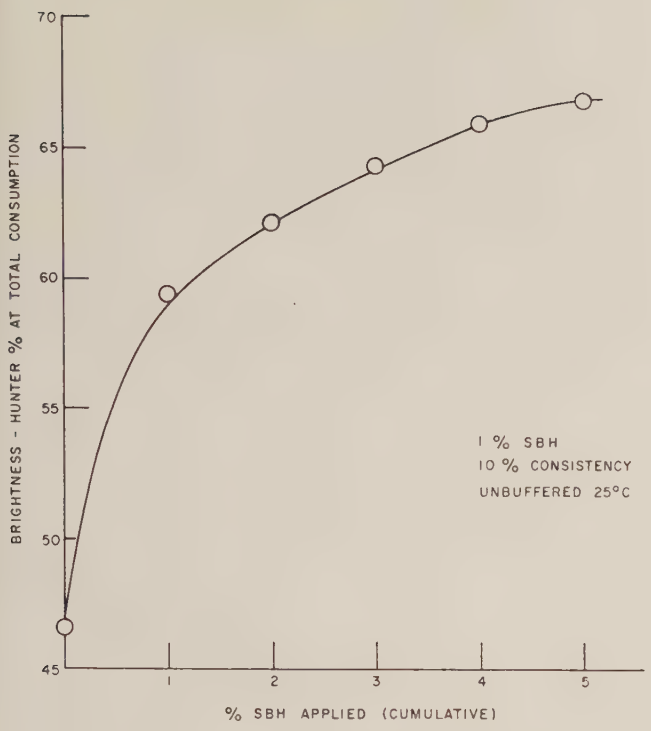

Fig. 1. Relationship between final brightness and applied chemical under unbuffered conditions

achieved if the reduced pulp from the first stage is further reduced with sodium borohydride.

The loss in brightness on reductive unbuffered bleaching can be attributed to the interaction of Fe^{+3} or other metals and sulfur (from neutralization with sulfur dioxide) in the pulps forming metal sulfides. However, prewashing with hydrochloric acid prevents this gray coloration from forming by the removal of the metal ions. The small additional brightness increment obtained in stage II suggests that further brightness increments are more difficult to attain. This is shown again in Fig. 1 where a cold soda pulp was successively reduced with 1% sodium borohydride at 10% consistency and 25°C., in each instance the bleach going to completion in 2 hr. It can be seen that the largest brightness increment occurs during the first 1% treatment with succeeding treatments having a diminishing effect. The same pattern is repeated when the reaction is done in 0.1 *N* NaOH as shown in Fig. 2. Comparing Fig. 1 with Fig. 2 it is seen that the same final brightness is achieved with 1% applied chemical after 48 hr. in the alkaline bleach as compared to 2 hr. in the unbuffered bleach. However, in the case of the alkaline bleach only 50% of the sodium borohydride was consumed. A similar relationship between brightness and chemical consumption is obtained when comparing the two types of bleaches with 2% $NaBH_4$.

These results show that a better utilization of chemicals is obtained under alkaline conditions (0.1 *N* NaOH) where the sodium borohydride is more stable. However, the brightness is developed more slowly under these conditions. The brightness development can be greatly enhanced by increasing the temperature to 75°C. as shown in Fig. 3. In fact, at a chemical consumption of 1% the same brightness value is obtained after 6 hr. at 75°C. as compared to 96 hr. at 25°C. under alkaline conditions. To obtain the same brightness under unbuffered conditions, 2%

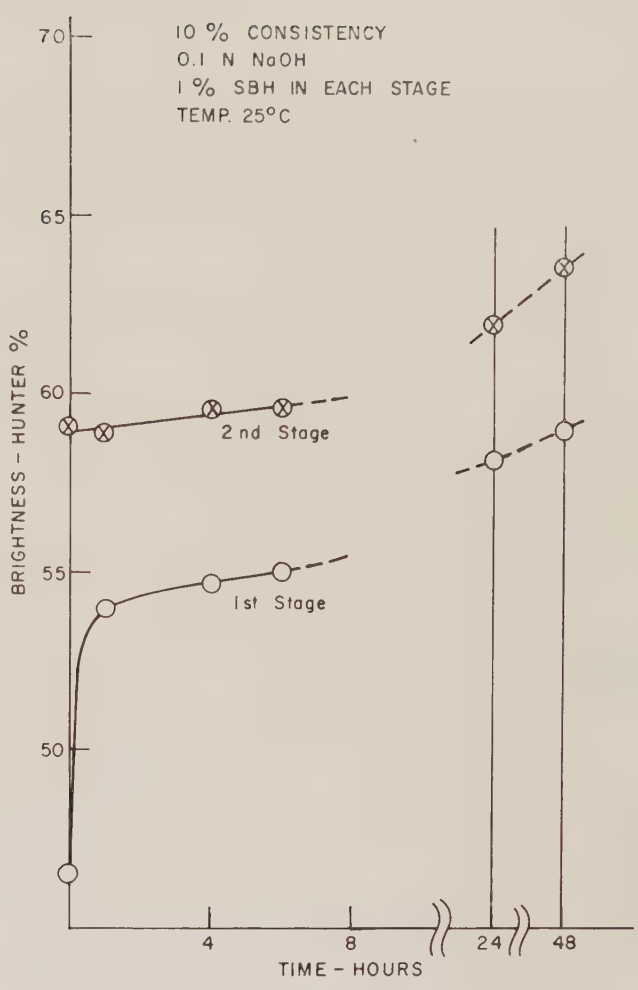

Fig. 2. Brightness development in two stages under alkaline conditions at 25°C.

chemical is required. The brightness obtained with 2% chemical at 75°C. under alkaline conditions is equivalent to five stages (5% chemical) under unbuffered conditions at room temperature.

In both the unbuffered and the alkaline reductions a fast increment in brightness is followed by a slower one.

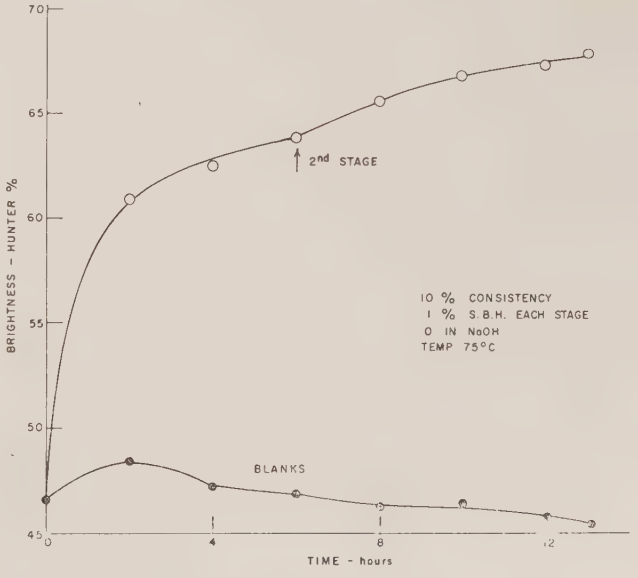

Fig. 3. Brightness development in two stages under alkaline conditions at 75°C.

may involve the reduction of two types of groups at different rates or the same group with different degrees of accessibility.

Factors Controlling the Rate of Brightness Development

In the sodium borohydride reduction of cellulose complete reduction is difficult to achieve (*24*). Recently, the more easily reduced groups in cellulose were ascribed to the more accessible carbonyl groups in the pulp (*25*).

The brightness increments observed in this work are ascribed to reduction of the noncarbohydrate material in the wood. Nevertheless, from the known distribution of these materials through wood (*26*) accessibility may play a role in determining the rate of reduction. Several types of experiments were therefore performed to determine the extent which brightness development may vary with accessibility.

A. Chemical Accessibility

The results of sodium borohydride bleaching thus far have been expressed in terms of brightness and amount of chemical used. These quantities do not give any information on the chemical alteration of the pulp due to treatment with sodium borohydride. The reducing power (copper number) of the pulp was chosen as the best available method to describe the chemical changes incurred to the pulp during reduction.

It is well known that the properties of cellulose can be altered by swelling in alkali of various concentrations. It was therefore reasoned that the chemical accessibility would be increased during reductions at higher alkali concentration due to a greater amount of swelling. Cold soda pulps over a wide range of alkali concentrations, 0.4 to 12% NaOH (by weight), were reduced with sodium borohydride. The brightness values and the copper number of the pulp in addition to the sodium borohydride consumed were determined. These are shown graphically in Figs. 4a and 4b. It can be seen that in the concentration range between 1 and 7% NaOH, the brightness increment and decrease in reducing power on reduction are about equal. In fact, a linear relationship was obtained between brightness and copper number in this alkali concentration range. Since the reducing power (copper number) of carbohydrates is not related to brightness, this relationship may be ascribed as originating from the lignin and other noncarbohydrate material in the cold soda pulp.

The rate of brightness development decreases slowly with increasing alkali concentration. However, in 12% NaOH the rate of brightness development and chemical reduction is greatly reduced. This may be due to the fact that in 12% caustic intramicellar swelling of the cellulose lattice occurs. This in turn would result in an enclosure of the lignin in a gellike material leading to reduced accessibility.

Another method of altering the accessibility is to reduce the pulp in solvents which swell fibers to varying degrees. Reductions were therefore done in a variety of solvents where sodium borohydride is soluble under a set of standard conditions. These were 3% consistency, 2% $NaBH_4$, 50°C., and a reaction time of 3 hr. Table II lists the results along with

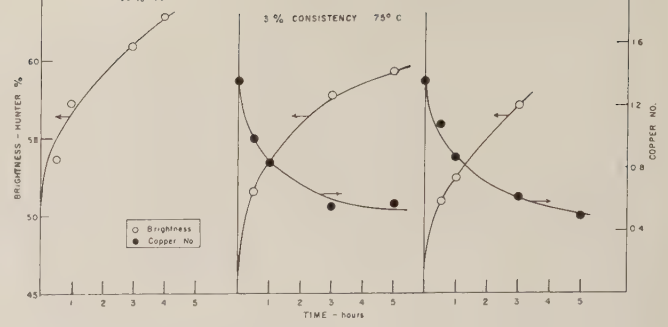

Fig. 4a. Brightness development and copper number at various alkali concentrations at 75°C.

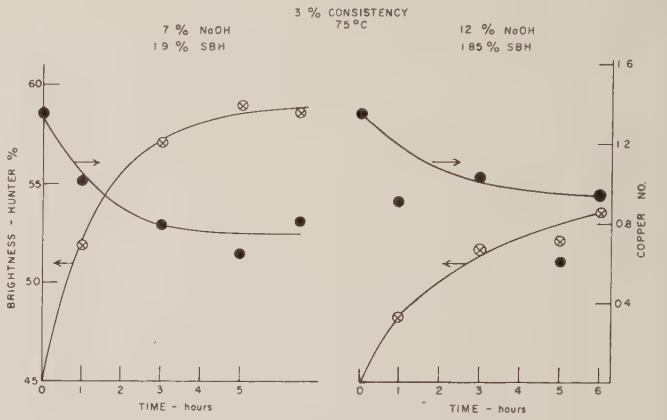

Fig. 4b. Brightness development and copper number at various alkali concentrations at 75°C.

the cohesive energy density values of the solvents. The results do not show any clear correlation between brightness attained and swelling capacities of the solvents. It can be further noted that the rate of reduction, e.g., brightness development is slightly faster in acetonitrile than in water.

On the basis of these results it is tempting to conclude that the sodium borohydride reduction of cold soda pulp is not affected by changes in accessibility induced by alkali or solvent swelling. However, in view of other changes such as increased charge effects in alkali which may be occurring simultaneously tending to retard the rate of reduction and a different reducing mech-

Table II. Reduction of Cold Soda Pulp in Water and in Nonaqueous Solvents

Pulp	*Solvent*	*Time*	*Brightness*	*Cohesive energy density*
I	Acetonitrile	3 hr.	67.0	11.9
I	Isopropyl alcohol	3 hr.	64.9	11.5
I	Tetrahydrofuran	3 hr.	64.1	11.4
I	Ethylene carbonate	3 hr.	62.1	14.7
I	Water	3 hr.	63.7	24.0
I	Water	0	47.5	..
J	Water	0 min.	46.5	..
		5 min.	56.0	..
		10 min.	56.0	..
		30 min.	57.6	..
		60 min.	57.6	..
		210 min.	56.6	..
J	Acetonitrile	0 min.	46.5	..
		5 min.	57.0	..
		10 min.	57.5	..
		30 min.	58.2	..
		60 min.	59.1	..
		150 min.	58.0	..

anism in nonaqueous solvents such a conclusion may not be warranted at this time.

B. Physical Accessibility

Since the sodium borohydride reduction of pulp is a heterogeneous topochemical reaction, it was of interest to study the effect of pulp freeness on the rate of chemical consumption and brightness development. Two pulps with Canadian Standard freeness values of 320 and 150 ml. and initial brightness values of 39.5 and 38.7%, respectively, were reduced with 1%

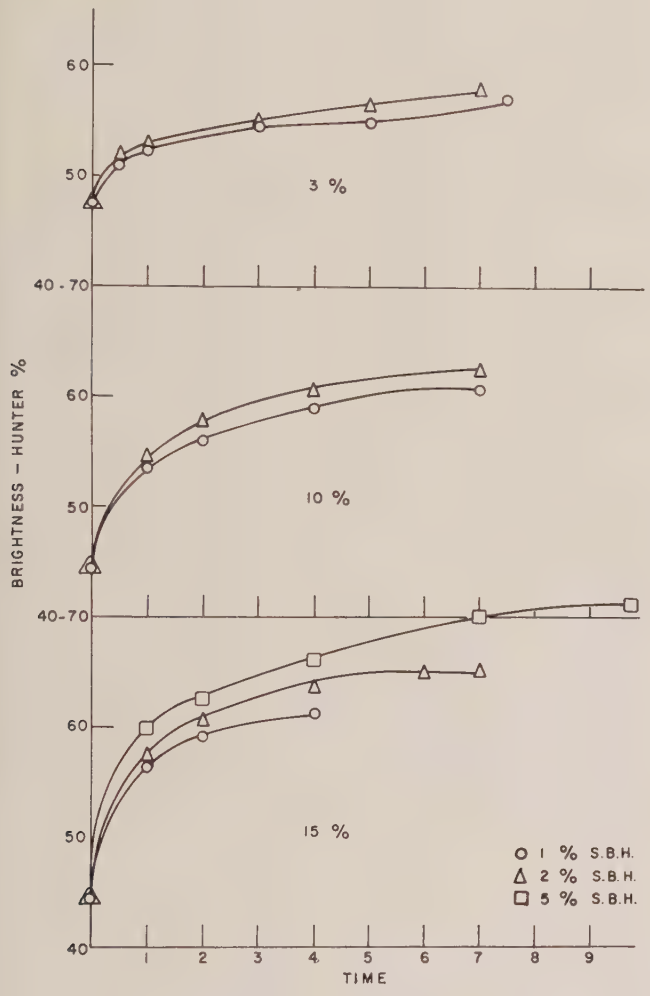

Fig. 5a. Rate of brightness development at 50°C. and 3, 10, and 15% consistency

$NaBH_4$ at 1% consistency and 35°C. in 0.1 *N* NaOH. In the first hour the chemical consumption was about equal, but at later stages the 320-ml. freeness pulp consumed 10% more chemical. The actual sodium borohydride consumption for reduction was obtained by subtracting that due to decomposition (20%) from the total consumption value. While not shown here, a plot of the brightness point increment against chemical consumption for the two pulps showed that the 320-ml. pulp gave a one-half point larger increment at 50% chemical consumption than the 150-ml. freeness pulp.

These results are somewhat unexpected in view of other findings (*34*) of an accelerated reaction rate due to smaller wood particle size. Evidently, freeness values cannot be used as a criterion for particle size or surface area available for the reduction reactions. With this limited data on explanation is offered for the apparent higher chemical consumption for the higher freeness pulp, but it would seem that already at 320-ml. freeness the maximum fiber surface area for reduction with sodium borohydride is reached.

Effect of Chemical Concentration and Temperature on Brightness Development

As indicated previously, reductive bleaching under

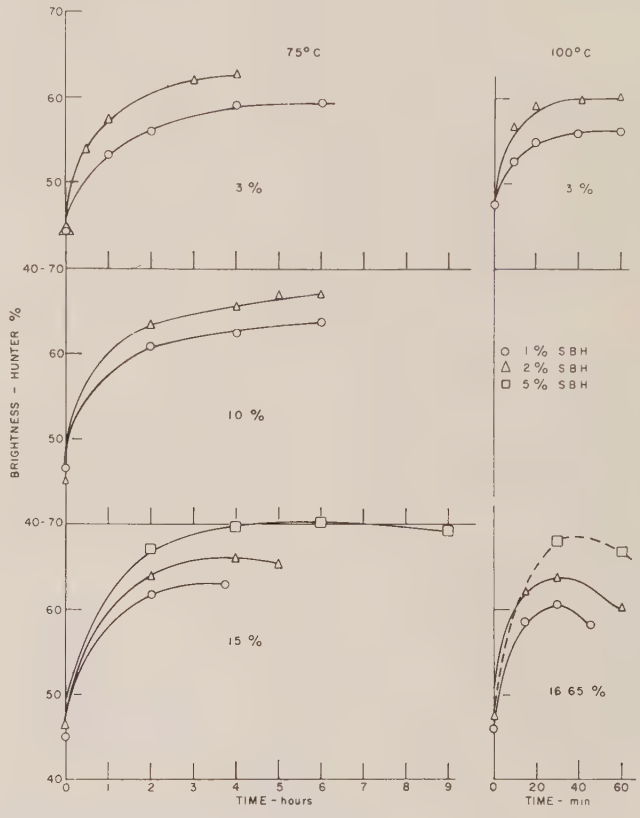

Fig. 5b. Rate of brightness development at 75 and 100°C. and 3, 10, 15, and 16.65% consistency

alkaline conditions at 25°C. is a slow process but, as shown, brightness increments are substantial when sufficient sodium borohydride is chemically consumed. Several variables affecting the brightness were therefore investigated. The amount of chemical employed was 1, 2, and, in some cases, 5% based on the weight of pulp. The temperature range investigated was between 50 and 100°C., while the consistencies were 3, 10, and 15%. These reductions were all done in 0.1 *N* NaOH.

At a given temperature, the results in Figs. 5a and 5b show that the brightness increases rapidly at first, followed by a further slow development. This continues as long as there is excess chemical. Increasing the sodium borohydride concentration from 1 to 2% has a greater effect at higher than at lower temperatures. With 1% $NaBH_4$ on the weight of the pulp, the brightness values after 4 hr. at 50°C. are 55, 59, and 61% corresponding to 3, 10, and 15% consistency. At 75°C. the brightness values for the same conditions are 56.5, 62.5, and 63% with no further brightness increments. No doubt a better utilization of sodium borohydride is achieved at 50°C. due to the lower

decomposition rate of sodium borohydride at the lower temperature. At 100°C. the maximum brightness develops in 30 min. after which reversion sets in. The maximum brightness achieved at 100°C. for 1, 2, and 5% $NaBH_4$ at 15% consistency is 60, 64, and 68%. At 50°C. for the same bleaching conditions the brightness values are 62, 65, and 71%. However, large differences in time are involved in achieving these slightly higher brightness values.

A number of bleaches were done under unbuffered conditions similar to those of Mayer and Donofrio (*22*). They found that 35°C. was the optimum temperature to achieve maximum brightness under unbuffered conditions. Experiments with cold soda pulp indicated that only a 10 to 12-point brightness increment could be obtained under unbuffered conditions irrespective of the temperature with 1 or 2% chemical and at consistencies between 3 and 20%. The results thus indicate that the most effective conditions for the reductive bleaching of cold soda pulp with sodium

Table III. Sodium Borohydride Reduction of White Birch Chips During Kneading. Ten-Min. Steep and 50-Min. Kneading

NaOH, g.p.l.	*Liquor: wood, l/kg.*	*$NaBH_4$ % o.d. wood*	*$NaBH_4$ consumed — %*	*$NaBH_4$ consumed — g./100 g. o.d. wood*	*Hunter brightness, %*
48.4	5:1	..	..	..	42.8[a]
52.5	5:1	2.16	26.6	0.575	58.6[b]
50.6	5:1	0.68	69.0	0.483	55.2[b]
47.3	2.5:1	0.62	96.5	0.600	53.7[c]
54.3	10:1	0.70	41.2	0.290	51.8[d]
47.9	2.5:1	1.94	84.8	1.65	62.9[d]

[a] Buchner handsheets.
[b] Sheet mold handsheets. Pulp screened and cleaned.
[c] Sheet mold handsheets.
[d] Buchner handsheets. Pulp screened and cleaned.

borohydride are high consistencies, between 10 and 20% relatively high temperatures, between 50 and 75°C., in suspensions containing 0.4% NaOH.

Effect of Salt on Sodium Borohydride Reduction

It has recently been reported (*35*) that a number of salts at pH 10.4 enhance the reduction of carbonyl groups in cellulose. Consequently, a few controlled experiments were done to see whether any catalytic effect could be observed in the reduction of cold soda pulp. A series of pulps at 3% consistency in 0.1 *N* NaOH were reduced with 2.12% $NaBH_4$ borohydride at 50°C. and 2% LiCl, NaCl, and KCl, respectively. Both the rate of development and the final brightness were the same as the controls. Since the catalytic effect was observed at pH 10.4, the effect of 4% Li, Na, and K chloride on brightness values was also tested on a cold soda pulp reduced unbuffered with 2% $NaBH_4$ at 3% consistency and 25°C. Again, the salts did not enhance the brightness of the pulps when compared to controls. Evidently the catalytic reduction effect observed for pulps is not associated with any brightness development.

Sodium Borohydride Reduction During Pulp Preparation

Several workers have shown that a good fiber separation is obtained mechanically when hardwoods are presoaked with sodium hydroxide (*27*, *28*). This is the basis of the cold caustic process. During this caustic treatment the wood becomes dark in color. The extent of the brightness loss depends on the wood species and alkali concentration. The reactions occurring to give the darker color are largely unknown, but are currently being investigated in these laboratories. The commercial value of the cold caustic proc-

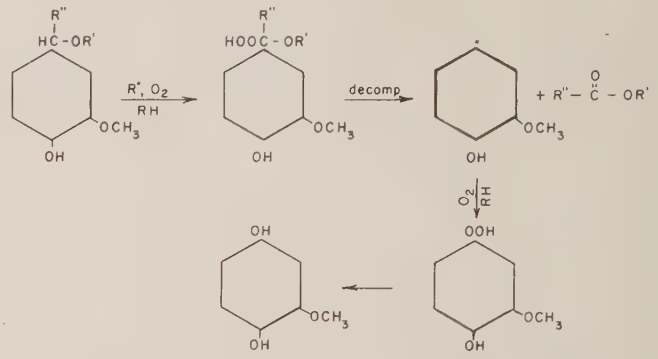

Fig. 6. Proposed mechanism for the formation of quinone derivatives by free radical attack on lignin

ess would be considerably more if these pulps would have higher brightness values.

Since it was found that cold caustic pulp can be bleached with sodium borohydride and the latter is stable in alkaline solution, it was thought that the dark color formed by the interaction of wood with alkali could be prevented in the first instance if the sodium borohydride were present in the caustic solution during the conditioning period.

White birch chips were soaked for 10 min. in a range of alkali and sodium borohydride concentrations. The chips were then disintegrated in a mechanical kneader for an additional 50 min. The pulp was then refined by a single pass through a disk refiner at −2 mils clearance. The initial and final concentration of sodium borohydride was determined. These results are tabulated in Table III. It can be seen that considerable brightness increments are obtained with initial sodium borohydride concentrations of 1 and

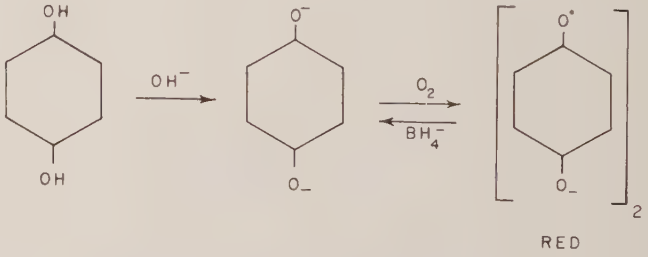

Fig. 7. Proposed mechanism for the color formation from quinone derivatives

2%. Analysis indicated that sodium borohydride was not completely consumed. It appears then that the caustic solution containing the reducing agent can be replenished and reused. These results indicate that the presence of a reducing agent under alkali conditions prevents the formation of deeply colored bodies. Since the previous work has shown that the bleaching response increased rapidly under alkaline conditions with temperature, refining the soaked chips

containing sodium borohydride in a disk mill may greatly accelerate the brightness development. This area of reductive bleaching remains to be explored.

DISCUSSION

To appreciate fully the chemical and brightness changes occurring to cold soda pulp on treatment with sodium borohydride a brief discussion of the origin of the coloring components in wood and particularly cold soda pulp is desirable.

The Origin of Color in Cold Soda Pulp from White Birch

Wood in the natural state can vary in color from practically white to black. Since the carbohydrate portion is white the coloring bodies are usually ascribed to the noncarbohydrate portion of wood, i.e., the lignin and extraneous matter. In deeply colored wood such as ebony the extractives can largely account for the deep color. However, even on complete removal of these extractives the wood still remains dark. In wood species that are lighter in appearance the obvious source of color is much more difficult to trace since the extractives may be low and not highly colored. Even in these cases extractions of various types do not remove the color significantly. Evidently part of the coloring components may be deeply embedded material incapable of being extracted but nevertheless capable of undergoing chemical reactions. This suggests that a portion of color-producing bodies are ligninlike materials or part of the lignin macromolecule itself. Considering the recent theories of Freudenberg (*29*) regarding the biosynthesis of lignin, it seems likely that quinones are present in the lignin molecule. These quinones may partly contribute to wood its natural hue and also account for the sensitivity of wood toward acids and bases.

Another possibility for the origin of quinones is through a free radical attack on the lignin by oxygen as shown by the series of reaction in Fig. 6. The role of oxygen in producing color changes is not an isolated case in the pulp industry. It has been reported a number of times that groundwood from stored wood has a lower brightness than green wood and also that waterlogged wood gives a higher brightness than the same wood when dried below the fiber saturation point (*30*). These results may be explained on the basis that lignin in wood reacts with oxygen to yield derivatives which are more colored or become colored by chemical interaction with alkali.

Further evidence that quinones are involved in producing color in white birch cold soda pulp was obtained when it was found that the characteristic red-brown color formed when the alkali and wood interact was absent when the wood and alkali were mixed under oxygen-free conditions (total pressure 1×10^{-4} mm.). Only a yellow-brown color persisted. These results may be explained on the basis that the semiquinone radical is formed in the presence of oxygen as shown in Fig. 7. The enhanced brightness obtained when wood or cold soda pulp is reduced with sodium borohydride can consequently be partially explained on the basis of reduction of the semiquinones.

The phenolic material that is liberated when wood is treated with hot or cold water or alkali is probably another source of color in cold soda pulps. In addition to tanninlike material (*31*), monomers such as vanillin, ferulic, and *p*-hydroxybenzoic acids and other guiacyl and syringyl containing units (*32*, *33*) have been found in alkali extracts. These monomers and tannins in the presence of air and/or base undergo condensation reactions with each other or with themselves giving dark colored solutions (*38*). Since these materials are strongly absorbed by the pulp they no doubt contribute to the color of wood or wood pulp. Spectral studies of these solutions have indicated both the phenolic and carbonyl nature of these solutions (*34*). Consequently, in acid solution, by decreasing the ionization and shifting the absorption toward the ultraviolet, the color of the solutions in the visible region is decreased. Sodium borohydride decreases the absorption in the visible region of these compounds by reducing the carbonyls to alcohol groups. This results in a much lower absorption at 350 μ, resulting in a decreased absorption in the visible region.

The reduction of the carbonyl groups in lignin with sodium borohydride as mentioned previously was studied by Gierer (*20*) and Adler (*19*). It is likely that the reduction of these carbonyl groups contributes to the total brightness increment as suggested by Mayer and Donofrio (*22*).

The rapid initial rate of brightness increment both at high and low pH values may be explained on the basis of a topochemical reaction, the initial high rate being due to the reduction on the available external surface while the slower rate, both at high and low pH values, is due to the necessity of diffusion into the fibers before reduction.

The slower rate of reaction in alkaline solution as compared to the unbuffered case at the same chemical concentration could result from the larger electrostatic repulsion between the BH_4^- anion and the cellulose at higher pH values. In addition, the rate of reduction of para-hydroxyl phenyl ketones or aldehydes are retarded when these are ionized at high pH values (*18*).

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Brightness of High Yield Pulps

I. Morphology and Nature of Coloring Materials in Wood

RENATA MARTON

High yield cold soda hardwood pulps exhibit a brown discoloration which is resistant to the action of strong bleaching agents. A microscopic examination of the morphological elements of the woods from which these pulps were prepared revealed that the brown discoloration was due to material located in the cells of the wood. In addition, it was observed that in the heartwood these substances are darker and more abundant than in the sapwood. Similar formations could be detected in the fibers and other morphological elements of a cold soda pulp prepared from the heartwood of white birch. Microspectrophotometry and other physico-chemical methods were used to characterize the colored incrustations in wood *in situ* and in the pulp fibers. The results suggest that the color of the high yield cold soda pulps from hardwoods is due mainly to the brown bodies in the morphological structure of wood. Chemically these incrustations are not identical with the lignin, but probably develop from the same precursors. Their behavior in bleaching is different from the response of lignin towards the conventional bleaching agents.

Mechanical and chemical energy are two forms of energy which when applied to fiberize wood in a variety of procedures produce different effects on the properties of the fiber element.

In mechanical pulping there is practically no dissolution of wood components resulting in groundwood—a pulp of the highest yield obtainable from the wood. The chemical pulping processes are based on the solubilization of all the incrustants of the wood, leaving cellulose in as pure a state as possible. The latter procedures lead to lower pulp yields than with mechanical pulping. Since the present trends in pulping have been toward a better utilization of the raw material, interest has been focused on processes where only a part of chemical incrustants is removed from the wood before or during the mechanical fiberization, thus improving the yield. These types of pulps are known as high-yield or semichemical pulps. The use of these pulping procedures has been stimulated by their successful application to hardwood species. Acid sulfite, neutral sulfite, sulfate, soda treatments have been applied for the chemical part of the production of the semidelignified pulps as well as various refiners for the mechanical side. The usefulness of the semichemical pulps, however, is limited because of their low brightness. Although it is observed that the acid and neutral sulfite pulps generally are brighter than the alkaline pulps, the semichemical pulps respond to bleaching quite differently than either chemical or mechanical pulps. In the bleaching of chemical pulps the relatively small amount of lignin (0.5 to 6.0%) left after pulping has to be removed or altered in order to reach a high brightness. In the case of mechanical pulps nearly all of the colored components of the original wood (lignin, extractives, etc.) must be treated.

The high-yield pulps occupy an intermediate place between the two extreme cases of pulping described above. They contain substantial amounts of the coloring materials, hence their removal to obtain high brightness by any means is out of the question, since the objective of a high yield is thereby defeated. Many bleaching agents have been tried on semichemical pulps (*1*), hydrosulfite, peroxide, hypochlorite, chlorites, and it has been shown that the different bleaching agents have quite different effects on the properties of the pulps. On the other hand, it was observed that there is an upper limit of bleachability which, for many pulps, is a serious shortcoming for their application in paper finishes. The unfavorable color balance has never been solved because the chemical changes which occur in bleaching high-yield pulps are different from those occurring when full chemical pulps are bleached.

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A further handicap of the semichemical pulps is their color reversion.

It also is interesting to note that the brightness characteristics of purely mechanical pulps from softwoods or hardwoods compare more favorably in color with the semichemical pulps and the color balance becomes intensified when the woods are chemically pretreated with alkaline liquors.

The question arises, to what can the dark discoloration of these products be due? Does it originate in the lignin? If lignin would account mainly for the dark color of high-yield pulps, it might be expected

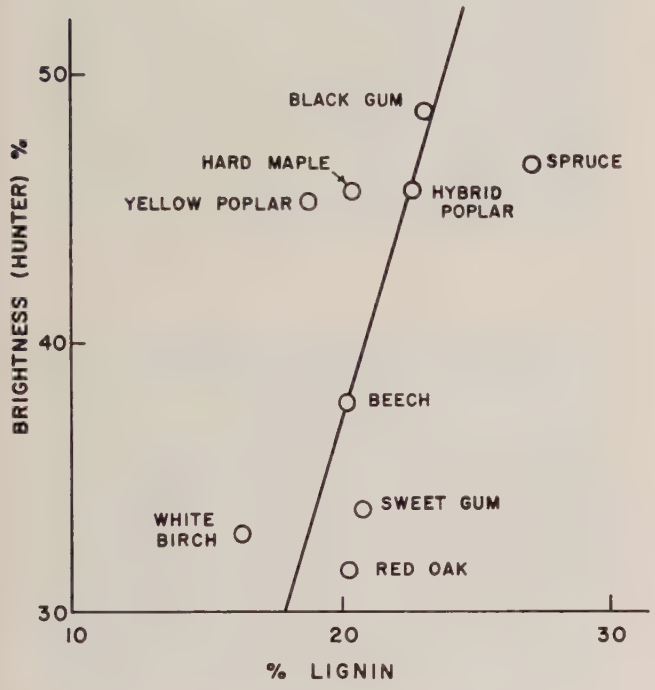


Fig. 1. Brightness of cold soda pulps from various woods related to their lignin content

that woods with higher lignin content would result in darker pulps than those with a lower lignin content when pulped by the same procedures. Figure 1, which represents the brightnesses of cold soda pulps prepared in the same conditions from woods with different lignin contents, gives evidence that this is not likely because, in many cases, a wood specimen with a lower lignin content results in a darker pulp than a stronger lignified wood. These data, however, are not an unambiguous proof that lignin does not determine the color of the pulp.

"Extractives" also have been considered as another possible cause of the dark color of high-yield pulps, because they may become highly colored as a result of chemical treatment despite low amount. Brightness improvement due to the extractions with various solvents was, however, so insignificant that the latter view had to be abandoned (*2*).

The coloration of cold pulps prepared from various hardwoods is so pronounced that it seemed obvious that microscopic observations would help to locate the colored substances in the fibers and by means of this technique elucidate their part in the pulping and bleaching.

Many fibers of cold soda pulp from whole birchwood when observed under the microscope showed an overall darker shade than others and, in addition, some of them contained brown droplike and bridgelike formations in the lumina which were not known as normal constituents of the cells. Since these constituents were pre-existent in the wood before the application of the pulping procedures, they had to be detectable in the wood itself. A look at sections of tree boles revealed great color differences between species and these differences were further accentuated in the same stem in the sapwood and heartwood parts.

EXPERIMENTAL APPROACH

Microscopic studies have been effected on unstained sections of various species, with emphasis on white birch, and on fibers prepared from these wood specimens. After the colored incrustations have been located in the morphological elements of the wood, their separation, removal, and identification can be attempted by the conventional methods. Microspectrophotometric techniques have been applied for the study of the colored components in the wood *in situ* as well as on the pulp fibers.

MICROSCOPIC OBSERVATIONS

Fiber suspensions of cold soda birch pulp have been observed under the microscope without staining. In the preparation the whole fibers were of two distinctive shades: some light cream, others of a pronounced brown tint. In addition to the color differences of the cell walls themselves, many of the dark-colored fibers contained brown outgrowths of various shapes and dimensions in their lumina (drops, columns with menisci characteristics for liquids, bridges, warts, etc.).

The ray cell fragments found in the preparations also showed lighter and deeper shades. Some of the vessels possessed orange brown tyloses, similar in color to the ray cell fillings. Much of the debris (fines) retained the color of the elements from which it originated. The study of pulps prepared separately from sapwood and heartwood fractions of the same bole revealed that the outgrowths are only in the heartwood fiber lumina. The darker shade of ray cells occurred also in the pulps prepared from the heartwood.

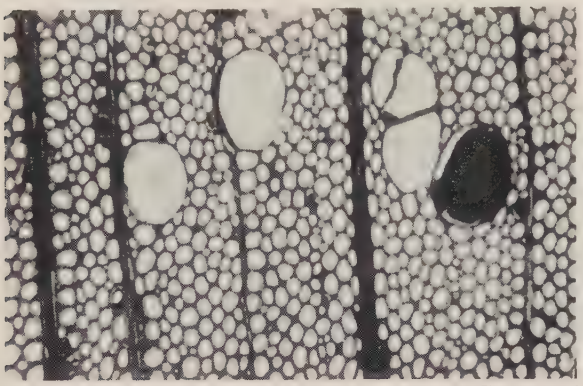
Tangential, radial, and cross-sections were prepared with a microtome, in 4, 10, 15, and 20 μ thicknesses from white birch and other species in order to find the location of these materials in the wood itself. The sections included the sapwood, the separation line, and a part of the heartwood zone. Slides mounted in water or in glycerine from the above samples were observed under the microscope *without staining*. It was found that the overall color of the sections was yellowish to brown and that the shade was darker in the heartwood part. The coloring materials were concentrated in the ray cells which were more abundant and darker in the heartwood (Fig. 2b). At higher magnifications other color sites were found: dark brown infiltrations in the lumina of the fibers appeared in the heartwood (Fig. 2c) and the vessels frequently were filled with tyloses of yellow to brown shades (Fig. 2a).

Extractions were carried out on microtome sections, on 200-mesh wood meal, and on pulp fibers in order to remove the coloring materials or to dissolve the incrusta-

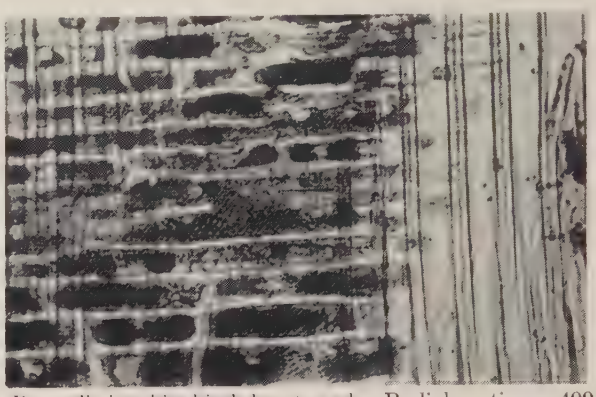
tions. The following solvents have been applied: water, ethanol, methanol, mixture of ethanol-benzene (1:1), mixture of methanol-benzene (1:1), ether, chloroform, acetone, methyl ethyl ketone, mineral acids, caustic soda, etc. Even after a long extraction the deposits showed under the microscope the persistent yellow-brown color and the shape of the membranes was not affected. The acid treatment brightened the shade; alkaline treatment darkened it to reddish-brown.

Ruthenium red and a combined treatment with Sudan III and haematoxylin changed the brown tint to dark purple in the fiber incrustations, while the deposits in the ray cells remained yellow.

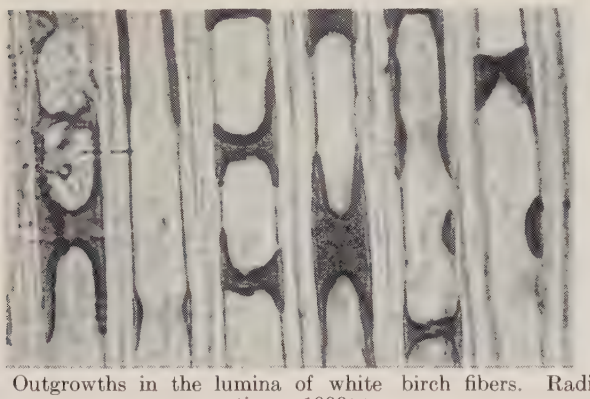
Microphotometric absorption techniques were used to study the components directly in the wood. This optical method leaves the observed material in its original state and permits the possibility of making measurements on very small particles (1 to 2 μ in diameter). In the present investigation spectra in



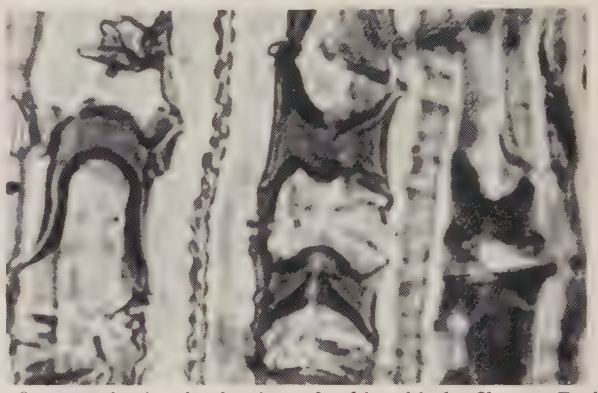
2a. Vessel tyloses in white birch heartwood. Cross-section. 300×.



2b. Ray cells in white birch heartwood. Radial section. 400×.



2c. Outgrowths in the lumina of white birch fibers. Radial section. 1000×.



2d. Outgrowths in the lumina of white birch fibers. Radial section treated with NaOH, 17.5%. 1000×.

2e. Fibers of cold soda pulp from white birch heartwood. 400×.

2f. White birch heartwood. Radial section treated with H_2SO_4, 72%. 200×.

Fig. 2. Photomicrographs of the colored substances in the heartwood and in the fibers of white birch

Staining. Numerous botanical and histological dyes known to have specific color reactions for cellulose, lignin, starch, resin, pectin, proteins, were used for identification of the material, but the result of these tests was inconclusive. Malachite green, however, stained the cell infiltrations deeper than the cell walls.

visible light have been recorded for vessel-tyloses, ray cell fillings, outgrowths in the fiber lumina and cell walls, in the natural state, and treated with caustic soda. The results given below are tentative and comparative, since the measurements were made on a relatively small number of cells, and it is obvious that a

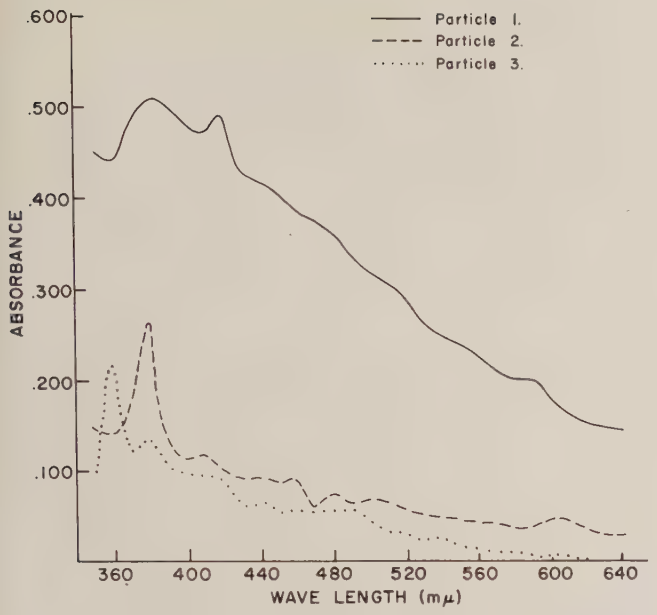


Fig. 3. Absorbance curves of various colored particles in the lumina of white birch heartwood fibers

great number of cells need to be investigated in order to obtain reliable quantitative results.

The experimental procedure was as follows: pulp fibers or cross or radial-sections of wood (4 to 10 μ thick) were embedded in water or in glycerol. The area of the element selected for observation was reduced by a special aperture to 4 μ^2 and the absorption of light was measured in the range 320 to 700 mμ. The method applied in this study is similar to the photo-

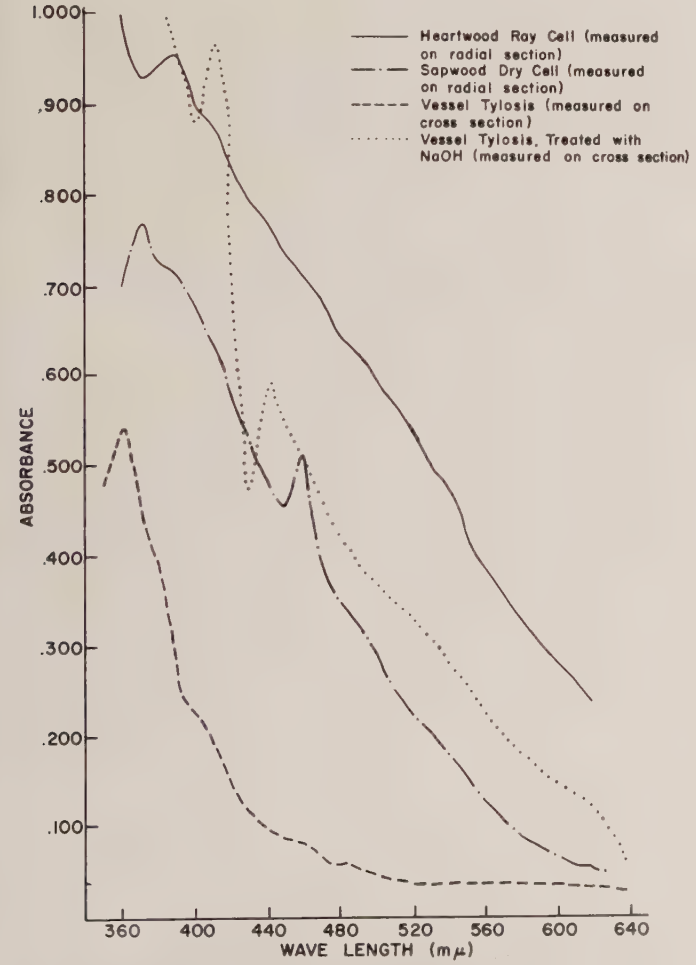


Fig. 4. Absorbance spectra of colored materials in ray cells and in vessel tyloses in white birch

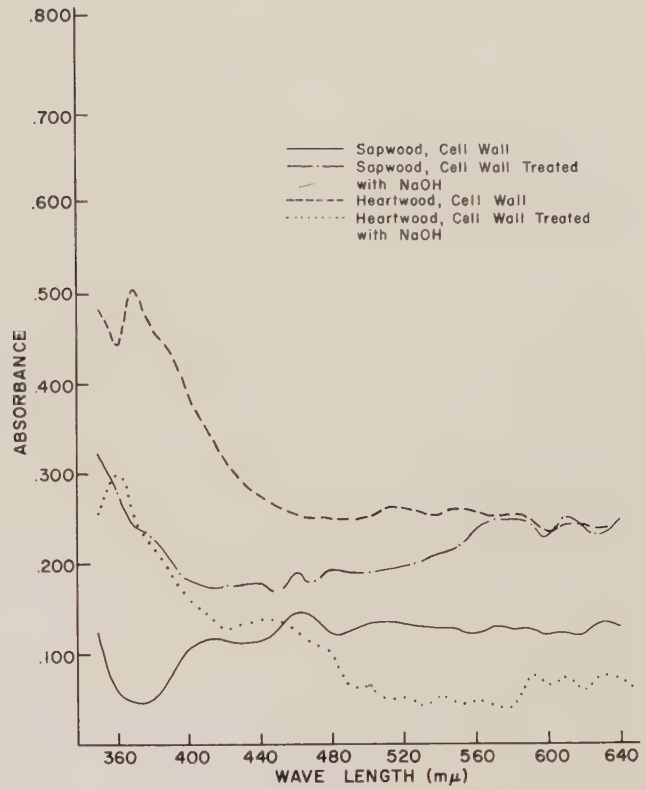


Fig. 5. Absorbance spectra of cell walls in white birch

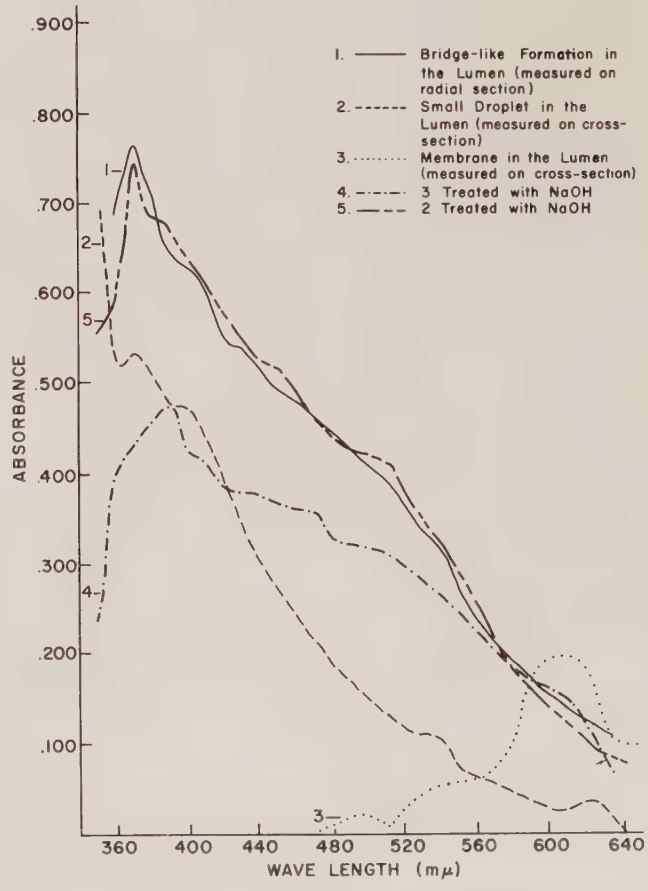


Fig. 6. Absorbance spectra of colored formations in the lumina of white birch heartwood

graphic method introduced by Lange (*3*) except that the absorption spectra of the fiber element under investigation are measured on an absolute scale. The apparatus employed for measurement *in situ* was a modification of one described by Pollister and Moses (*4*, *5*). The microspectrophotometer used in this study consisted essentially of a light source, a monochromator, or filter system for the isolation of narrow wavelength bands, a microscope, an arrangement for the projection of the image onto a diaphragm behind which is placed a sensitive photocell, usually a photomultiplier, and equipment for measuring the signal produced by the photocell.

The adsorption spectra of the colored particles in the wood cells have been recorded and are presented in Figs. 3, 4, 5, and 6.

Swelling and dissolution of the bulk of the cell wall was produced with cupriethylenediamine (cuene). Even a strong solution of cuene did not attack the linings of the ray cells, the tyloses, or the infiltrations in the lumina. The latter separate from the inner layer of the fiber wall maintaining their membrane shape. Similar effects were obtained when the sections of wood were treated with caustic soda of a concentration from 5 to 17.5% (Fig. 2d) or with concentrated sulfuric acid (72%) (Fig. 2d).

Isolation of the Chemically Resistant Colored Membranes. Heartwood was ground in a Wiley mill, the 200-mesh fraction screened and delignified with phenol and concentrated hydrochloric acid (*6*). The solubilized lignin was removed by washing with ether and ethanol. Following this treatment remaining cellulostic material was hydrolyzed with 72% H_2SO_4 for 2 hr., diluted—and boiled for 4 hr. (*7*). The brown residue was filtered and washed with water. It was insoluble in ethanol, hexane, and aqueous sodium hydroxide. It appeared as membranes under the microscope; some of the membranous material maintained the same shape as they had in the cells from which they originated (e.g., ray cells). The procedure also was tried in reverse order, i.e., the cellulosic material was hydrolyzed first by the standard procedure and then delignified. The residue was similar to that obtained in the previous treatment.

a. An x-ray diffraction picture was taken from the residue but no crystallinity could be observed. A pale halo obtained on the film indicated that the investigated material is amorphous.

b. An infrared spectrum also was prepared from the same sediment in order to detect the characteristic groups and attempt the identification of the material responsible for the color of the heartwood. The general aspect of the curve was similar to that of native lignin, though many bands were shifted toward the longer wavelength, some peaks were missing, and new ones appeared. The sharp OH stretching band at about 3 μ appeared as a broad and shallow band in the case of the investigated specimen. In the 3.42 μ range a peak indicated the presence of CH_2 groups, and at 6 to 7 μ a pronounced aromatic band group could be seen in the spectrum. Unidentified peaks appeared at 7.6 to 9 μ.

It should be recognized that the isolation of the residual membranes was effected by very drastic chemical means (phenol + HCl, H_2SO_4, etc.) many structural changes could have occurred in these processes, and therefore caution must be observed in the interpretation of these tentative results.

DISCUSSION

By the various microscopic techniques applied in the investigation of unstained water, or glycerine-mounted sections of wood and pulp preparations, it was established that yellow, brown, and reddish-brown materials are deposited in the cells of the wood and that these are to a great extent responsible for the coloration of the wood and for the color of the high-yield pulps prepared from the respective woods. From the collected observations, it can be concluded that, in the sapwood, the coloring materials of a pale shade are localized mainly in the parenchyma ray cells, while in the heartwood they are dark brown and can be found in greater abundance in the ray cells, in the vessel tyloses, and as bridges, droplets, warts, and columns plugging the lumina of the fibers. In the latter case, the occlusions appear to be bordered by a membrane which separates from the lumen wall as independent entities when the wood sections are swollen in cuene or in caustic soda. These materials already have been signalized by Pew (1949) (*7*) in his pioneering work on western hemlock and on groundwood. The optical and chemical behavior of the formations in the cells may be compared to that of the warts described by Wardrop, Liese, and Davies (*8*) and by Côté (*9*), who stated that "the warts are seen to be in form of diverticula or pouches containing a spherical object of great density toward electrons. It may be noted that where one of these diverticula is broken away from the membrane a hole is left so that the membrane is not continuous over the bases of the diverticula."

On the other hand, coloring materials can be detected in the wall (S_2 layer) of sapwood fibers, although in a very small amount, and in quite considerable quantities in the secondary wall of the heartwood fibers. Furthermore, the nature of the colorant in the cell walls is of approximately the same character as that of the ray cell occlusions, vessel tyloses, and plugs and warts in the fiber lumina.

The chemical reactions of the colored infiltrations in the heartwood provide sufficient evidence to prove that they are not formed of lignin, but that they are, nevertheless, somewhat similar to its nature and are derived from the same precursors as those which lead to the formation of lignin in plants.

The fact that sapwood does not exhibit brown bridges, columns, and warts in the fiber lumina and tyloses in the vessels indicates that these arise in the metabolic processes in the tree and in the formation of heartwood. From a plant physiology standpoint, the transformation of the living sapwood into "dead" heartwood occurs when the living cells lose their protoplast, i.e., when the sap, water, and food materials are withdrawn (*10*). Instead, the wood becomes infiltrated with various organic substances as it ages. Some of these substances impregnate the walls, others enter the cell lumina, and tyloses, if characteristic of the wood, are formed. That these phenomena are due to an enhanced metabolism—an increased physiological activity in the intermediate zone in front of the heartwood boundary (*11*, *12*)—was disproved by Frey-Wyssling (*13*). He demonstrated experimentally that

correlated with a diminished respiration activity of the living wood parenchyma. Frey-Wyssling further assumes that phenols (site of formation unknown) diffuse into the cell walls of the heartwood where they polymerize to insoluble dyestuffs.

A further contribution to the knowledge of formation of coloring materials in woody morphological elements was made by the studies (*14*) by Wardrop and Davies on lignification in higher plants. According to these authors when lignin is formed in living cells from eugenol, this artificial eugenol-lignin encrusts the cell walls and is, as with natural lignification, deposited within the cell wall between the microfibrils. However, the products of the lignification of eugenol are not uniform across the whole plant sections but differ depending upon the site of the formation. These authors prove that cytoplasm is not essential for the reaction to proceed in tissues. Because there is no evidence that the natural lignification will proceed in dead tissue (*15*), it can be assumed that eugenol in heartwood will be converted into a different compound than the eugenol-lignin formed under normal conditions. The view that lignin is formed in an oxidizing environment also is supported by Frey-Wyssling's theory of inhibited respiration of wood as the cause of heartwood formation.

Moreover, according to Adler's (*15*) interpretation of the reaction of lignification of the eugenol (conversion of the latter of coniferyl alcohol and then to coniferyl aldehyde) a highly reducing environment could possibly prevent oxidation of the eugenol which such a scheme demands. From the works of Wardrop, Frey-Wyssling, Fruedenberg, it is further apparent that lignification is a vital, not a passive process and thus may occur only in living cells. In the dead cells of the heartwood the transformation of such potential lignin precursors as coniferin and syringin follows a different pattern and leads to compounds of which the chemical behavior is different than that of the true lignin. These materials are more resistant than lignin is toward the conventional chemical reagents. Their location in the wood cells makes them also inaccessible for a mechanical elimination, handicapping thereby the bleachability of the high yield pulps prepared by the cold soda process.

It can be hoped, however, that investigation directed toward the chemical identification of the colored substances, which are underway in these laboratories, will help to overcome this very important difficulty.

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A Dynamic Stiffness Tester for Paper

F. T. RATLIFF

A dynamic, pendulum type stiffness tester for paper has been built. The instrument is capable of reproducibly testing the stiffness of a wide variety of papers. Because of certain features of its construction it is easier to use than its evolutionary predecessor, the torsion-wire pendulum stiffness tester.

A TORSION-wire pendulum tester was used by the staff of The Institute of Paper Chemistry to obtain fundamental information on the stiffness of a series of papers (*1*). This instrument had been earlier described by Van den Akker (*2*). The torsion pendulum was used by McPherson (*3*) in studying the effect of glycerin on paper, and by Nethercutt (*4*) to calibrate a sonic vibration tester for obtaining data on the stiffness of individual fibers. The original torsion-wire instrument was rather delicate, and gave some trouble from broken wires, and the like. E. H. Shriver (*5*), of the Mead Corporation Research Laboratories, built a "wire-less" torsion pendulum stiffness tester in which the entire restoring force is supplied by the sample itself. All of these instruments had a common operational disadvantage in that it is difficult to determine, with accuracy, the period of a pendulum whose oscillation is strongly damped. With some papers, the motion would die completely after only a few oscillations, making an accurate measurement of the period quite difficult. Dearth and Van den Akker (*6*), at The Institute of Paper Chemistry, further improved the torsion pendulum tester by mounting the entire instrument on a pivot so that it could be oscillated about the axis of the pendulum in a plane perpendicular to that axis. With this arrangement the oscillation could be made continuous by manually compensating for the damping effect with properly

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timed, small movements. This could not change the period of the pendulum, and improved the precision by enabling the operator to time many more complete oscillations than previously.

The tester shown in Fig. 1 combines the features of those built by Shriver, Dearth, and Van den Akker. The dynamic stiffness tester consists of a vertical-axis pendulum arrangement fixed by pinions between miniature ball bearings in a rigid assembly. One end of the test specimen is clamped to the pendulum with the clamp line coinciding with the axis of rotation. The free end of the specimen is restrained

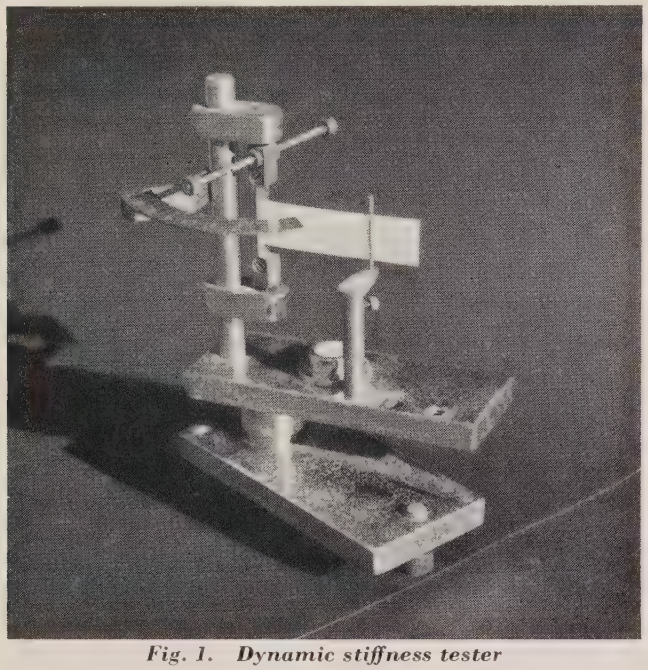

Fig. 1. Dynamic stiffness tester

by a wire slot placed some distance away. The pendulum has arms on which auxiliary weights may be placed. The whole mechanism is set on a vertical thrust bearing located at the axis of the pendulum to provide a means for supplying a manual "kick" to maintain periodic oscillation. The device is analogous, in principle, to the balance wheel of a watch. The paper sample provides the torque which acts as a restoring force to the pendulum and, together with the constants peculiar to the pendulum, fixes the period of oscillation. The manual force applied to maintain periodic oscillation is analogous to the main spring and escapement of a watch. As such, this force compensates for instrumental friction and internal friction of the test specimen and has no effect upon the period of oscillation. If the "kick" is applied out of phase with the natural period of the system, oscillation will cease.

The stiffness of the test specimen is expressed as flexural rigidity and is calculated according to the equation:

$$(EI') = 4\pi^2 I(b/3)\,(1/T)^2$$

where

(EI') = flexural rigidity in dynes, sq. cm.
I = dynamic moment of inertia of pendulum, gm. cm.^{2}
b = specimen length, cm. (distance from axis to restraining slot).
T = period of oscillation, sec.

The range of the instrument may be changed by varying the sample width or length or by adding auxiliary weights (in this case, small brass nuts) to the pendulum arms to change its dynamic moment of inertia. Illustrative of this latter change are moments of inertia calculated for several weight arrangements:

Weight arrangement	*I, gm. cm.*^{2}
No weights	55.75
One nut on each arm	107.32
Two nuts on each arm	154.04
Three nuts on each arm	195.82

The method for calculating I is shown in the Appendix. It is appropriate to express the flexural rigidity in terms of a unit width. In keeping with consistent units, the calculated (EI') should be divided by the sample width in centimeters.

The test procedure is simple. The instrument is made level, using the screws provided for the purpose. A sample of appropriate width and length is clamped in the jaw of the tester. The system is set into and kept in oscillation manually, using the instrument scale to maintain a reasonably constant amplitude of swing. The period is determined by timing a number of complete oscillations. Using the constants provided and the above equation, the flexural rigidity per centimeter of width is calculated.

Table I. Stiffness Tests on Several Papers

Paper	*Flexural rigidity (EI'), dynes—sq. cm./cm. of width* 1-ply	2-ply	4-ply	6-ply	8-ply
24-lb. plasticized glassine	...	...	164	...	154
22-lb. opaque glassine	...	...	214	236	...
25-lb. tracing	...	379	346	...	...
30-lb. glassine	...	626	611	...	..
40-lb. glassine	1340	1300	1330	...	..
50-lb. glassine	2830	2850	2730	...	...
Sulfite bond	2750	2610	2690	..	...
40-lb. wet-strength sulfite	3580	3820	...	...	...
Glassine-foil laminate	115,700	...	...	...	...

When measurements were first attempted, it was found that satisfactory oscillation could not be obtained with light weight plasticized glassine. If several plies of glassine were clamped in the tester, good pendulum motion was obtained. Theoretically, if the samples are clamped only at one end, and are free to slide over one another, the stiffness effects should be additive. This was checked on several samples of paper. In Table I, the flexural rigidity as determined from several sample arrangements is shown. The values obtained on multiple ply testing are sufficiently close to those obtained from testing single plies to justify the use of several plies when necessary.

The accuracy of the instrument depends primarily on the ability of the operator to time the oscillations with a stop watch. This error can be minimized by timing a sufficiently large number of cycles. When timing 20 cycles and making three observations per specimens, 95% confidence limits of ±2% for the average of a single sample can be expected. This means, of course, that sample width should be accurate to within ±0.025 cm. if precision is to be maintained.

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APPENDIX

The period of a torsion pendulum, T, may be expressed:

$$T = 2\pi\sqrt{I/K}$$

where

I = dynamic moment of inertia.
K = restoring torque per unit angle.

This equation may be rearranged to give:

$$I = T^2K/4\pi^2$$

If the moment of inertia of the system is changed by adding or removing weights we will have two equations, using subscripts 0 and 1 for different conditions:

$$I_0 = T_0^2K/4\pi^2 \quad \text{and} \quad I_1 = T_1^2K/4\pi^2$$

Rearranging:

$$K/4\pi^2 = I_0/T_0^2 = I_1/T_1^2$$

or

$$I_0 = I_1 (T_0^2/T_1^2) \qquad (1)$$

When the moment of inertia of a body is changed by adding weight, the new moment may be calculated by:

$$I_1 = I_0 + I_w + md^2$$

where I_w is the moment of inertia of the added weight taken through a central axis which is parallel to the axis of the whole system; m is the mass of the added weight; and d is the distance from the axis of the added weight to the axis of the system.

The brass nut weights can be considered as hollow circular cylinders, giving I_w the value:

$$I_w = m\,[(r_1^2 + r_2^2)/4 + L^2/12]$$

where r_1 and r_2 are inner and outer radii and L is length of weight (thickness of nut). We then have:

$$I_1 = I_0 + \{m[(r_1^2 + r_2^2)/4 + L^2/12] + md^2\}$$

Since the term in curly brackets will not change for any given arrangement of weights, we can say:

$$I_1 = I_0 + C \qquad (2)$$

If this equation is combined with (1) above:

$$I_1 = I_1 (T_0^2/T_1^2) + C \qquad (3)$$

or

$$I_1 = C/[1 - (T_0/T_1)^2]$$

The dynamic moment of inertia can be determined by measuring the period of the pendulum without and with auxiliary weights, using the same test specimen.

The dynamic moment for no added weight was calculated, using the average of 12 different determinations of $1/[1 - (T_0/T_1)^2]$ in equation (3).

This gave the value of 55.75 which was shown earlier.

The Effect of Pigment Binders on Paper Gloss, Printed Gloss, and Oil Absorption

ROBERT E. WEBER

This report is a study of the interrelationships of paper gloss, printed gloss, and oil absorption as it pertains to the adhesive concentration in paper coatings. The various adhesives studied along with a clay pigment are: (1) hydroxyethylated acid modified cornstarch, (2) corn white dextrin, and (3) soy protein. These coatings were applied by a KCM roll coater. The results show that printed gloss and oil holdout are improved with increased adhesive concentrations. Maximum printed glosses and oil holdout were reached at 36, 36, and 45% adhesive concentration, respectively (based on a weight ratio of adhesive to 100 parts of pigment) for the hydroxyethylated starch, soy protein, and white dextrin. At higher adhesive concentrations there were no changes in the above properties. The calendered paper glosses were found to decrease at increased adhesive concentrations. Minimum paper glosses were found to exist at the binder levels where the maximum levels of printed gloss were obtained. At higher adhesive concentrations, the calendered paper glosses were found to increase. The above findings are explained in terms of the void volume concept for pigments and by the varying degrees of adhesive bonding.

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THE PURPOSE of this study was to investigate the interrelationship of paper gloss, printed gloss, and oil absorption as it pertains to the adhesive concentration in clay pigmented coatings. The adhesives considered in this investigation were as follows: (1) acid modified hydroxyethylated cornstarch having 2% hydroxyethyl ether by weight, (2) corn white dextrin, and (3) soy protein.

To explain the findings of this report, it was necessary to consider a volume relationship of adhesive and pigment. Normally a weight ratio relationship of adhesive to pigment is used for paper coatings, but this proved to be less suitable for explaining the interrelationship of paper gloss, printed gloss, and oil absorption. The volume concept is not new. It has long been used by the paint industry, and more recently it was introduced to the paper coating industry by a number of investigators (*1–4*).

OIL ABSORPTION TEST

For studying the oil absorption properties of the various coatings, an oil absorption test based on the

transmission of diffuse light was used. Previously, this test was reported in detail by The Institute of Paper Chemistry (*5*, *6*), and these procedures were followed for the oil absorption test.

In brief, the principle of the test is as follows: a film of oil placed on a sheet of paper will render the sheet more transparent as the oil penetrates. Therefore, light that is incident to the sheet will become more readily transmitted as the oil penetrates the paper. The use of this type of transmission data along with the exponential law of absorption gives a good empirical approach for a homogeneous sheet.

The form of the exponential equation used for the oil absorption test is:

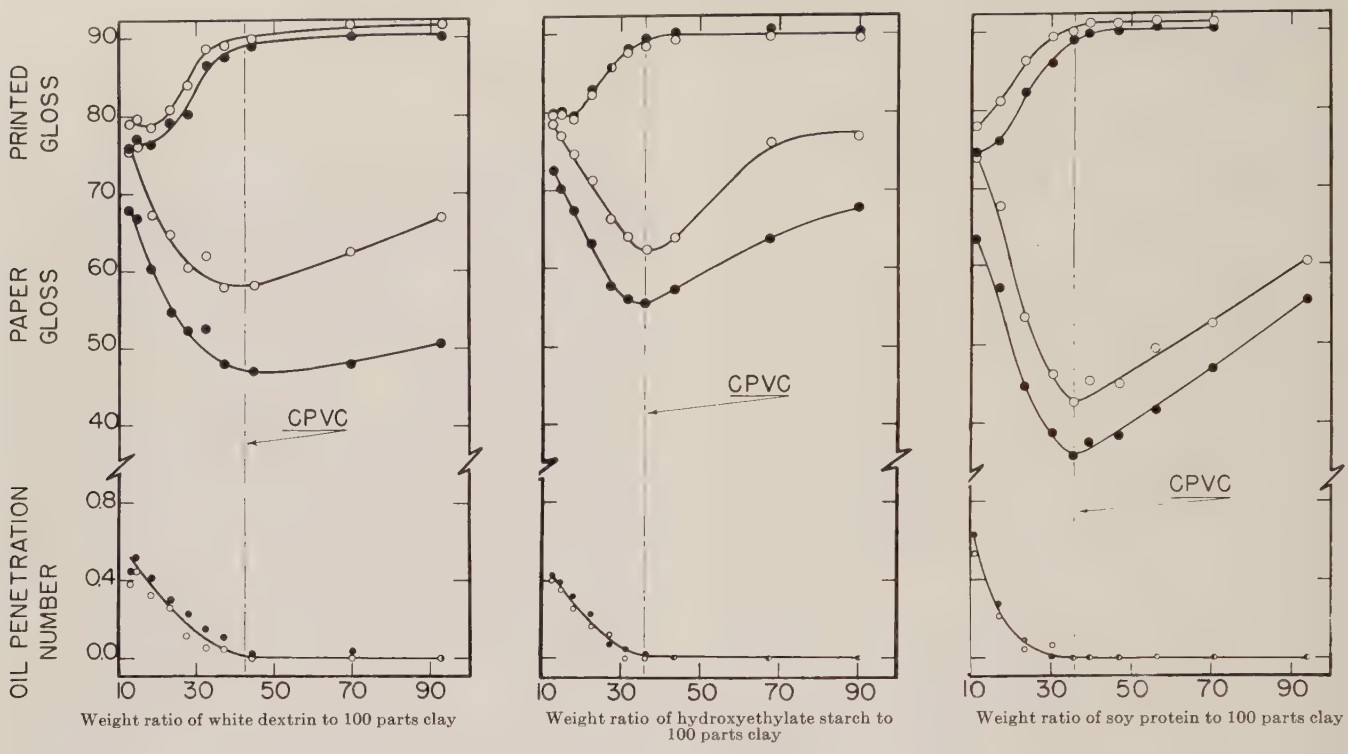
$$\frac{\log T - \log T_0}{\log T_\infty - \log T_0} = f$$

where

T_0 = initial transmission of light through the sheet
T = transmission after a given time interval of oil absorption
T_∞ = transmission after the paper is completely saturated by the oil
f = fraction of the sheet penetrated

However, a coated sheet of paper is not homogeneous. Therefore, a numerical answer, for example, of 0.20 does not truly represent an oil penetration of one-fifth the distance through the sheet. Likewise, a value of 0.50 does not necessarily represent half the sheet being penetrated by the oil. Because of this, the oil absorption values in this report are called "oil penetration numbers" rather than fractions of the sheet penetrated. Nevertheless, the "oil penetration numbers" are considered quantitatively significant in giving a good approximation for the degree of oil penetration.

The "oil penetration numbers" of this report are for 10-min. periods with a no. 0 litho varnish (linseed oil base). A paper coating with a zero "oil penetration number" for the above conditions is considered to be a continuous film for purposes of this study. This means the sheet is sealed against the passage of large molecular systems such as the vehicles found in oil-based inks.


PRINTED GLOSS
PAPER GLOSS
OIL PENETRATION NUMBER
CPVC
CPVC
CPVC
Weight ratio of white dextrin to 100 parts clay
Weight ratio of hydroxyethylate starch to 100 parts clay
Weight ratio of soy protein to 100 parts clay

Figs. 1, 2, and 3. Printed gloss, paper gloss, and oil penetration vs. the weight ratio of adhesive to 100 parts clay for the corn white dextrin, acid modified hydroxyethylated corn starch, and soy protein systems

The solid and open circles are papers that have been supercalendered 2 and 5 nips, respectively. The "oil penetration numbers" are 10-min. values with a no. 0 litho varnish.

EXPERIMENTAL PROCEDURE

It is well known that the properties of coated papers are influenced by many variables during manufacture. For this reason, most of the coating variables were carefully controlled in order to give a clear picture as to how the adhesive concentration influences paper gloss, printed gloss, and oil absorption. The variables that were considered constant for these experiments are as follows: (1) base paper, composition, and weight, (2) coating pigment, (3) coating preparation, (4) weight of coating, (5) coating equipment, (6) coated paper moisture at time of supercalendering, (7) supercalender temperature and pressure, and (8) composition and density of ink film for printing. The variables that could not be controlled because of the wide range of adhesive concentrations are: (1) coating solids and (2) coating viscosity.

Three clay pigmented coating series were prepared by using the following adhesives: (1) acid modified hydroxyethylated cornstarch having 2% hydroxyethyl ether by weight, (2) corn white dextrin, and (3) soy protein.

The coating formulation for the starch adhesives was as follows: 100 parts by weight (p.b.w.) of predispersed premium domestic kaolin clay, 1.5 p.b.w. of soluble soap, 0.8 p.b.w. of sodium hexametaphosphate, 0.3 p.b.w. of sodium hydroxide, and the ap-

propriate amount of the adhesive. As shown in Figs. 1 and 2, adhesive concentrations covered the range of 12 to 93 parts (moisture-free basis) for 100 parts clay. The starches were cooked at 95°C. (25% solids) for 10 min. and then mixed with the above components. The mixing was carried out on a Lightnin' mixer (model V7) for about 30 min. The total amount of solids per batch of coating was approximately 400 g.

The formulation for the soy protein series was the same as for the starch series with two exceptions: (1) one part of calcium stearate replaced the soluble soap and (2) for every gram of protein, 0.1 ml. of 29% aqueous NH_3 solution was used for dissolving the adhesive. As shown by Fig. 3, the adhesive concentrations covered the range of 11 to 94 parts (moisture-free basis) per 100 parts clay. In preparing these coatings, the protein was first soaked in water at room temperature for 5 min. The aqueous NH_3 solution was then added and the mixture was allowed to stand for an additional 5 min. (20% solids). The mixture was then heated to 50°C. and held at that temperature for 10 min. to insure complete dissolving of the protein. The other coating materials were then added to the dissolved protein and mixed in the same manner as for the starch coatings.

These coatings were applied to paper that was previously coated. The composition of this coated base stock was a 48 lb. base sheet of chemical fiber with 12 lb. coating, 6 lb. per side (25 × 38—500 ream size). The pigmented base coating was a starch-clay base coating used for a letterpress paper. The base-coated paper was used to provide a more uniform surface for the coatings to be tested. The test coatings were applied only to the wire side of the above base stock by means of a 12-in. KCM roll coater running at 140 f.p.m. The coat weights were adjusted to 6 lb. per ream with generally less than a pound of variation. The coated papers were then conditioned at 50% R.H. and supercalendered using 1, 2, 3, 4, and 5 nips, respectively, with the paper being reconditioned at 50% R.H. after each nip on the calender stack. The stack conditions were 593 lb. pressure per lineal inch with alternate steel and blue paper filled rolls. The roll temperature was approximately 160°F. The test sheets were always calendered with the coated side against the steel roll of the calender stack.

Oil penetration experiments were run on the test coated papers as discussed in the section, "Oil Absorption Test."

All gloss measurements were made with a 75° glossmeter with glossmeter tolerances as recommended by Zabel and Wink (7). The paper gloss values are averages of ten samples taken from a given experimental roll of coated paper, whereas, the printed gloss values are averages of six samples each. The printed glosses are obtained from solid prints that were made under controlled ink film conditions on an ATF Little Giant press. The ink used for these tests was Interchemical Corp.'s blue gloss testing ink.

RESULTS AND DISCUSSION

Printed Gloss and Oil Absorption

The results show that oil holdout and printed gloss for oil-based inks are improved with increased adhesive concentrations. There is a direct correlation between the printed gloss and oil absorption properties of the coatings. The results are shown in Figs. 1, 2, and 3. The minimum amounts of adhesive to produce maximum printed gloss are 36 ± 2, 36 ± 2, 45 ± 2%, respectively (based on a weight ratio of adhesive to 100 parts pigment) for acid modified hydroxyethylated cornstarch, soy protein, and corn white dextrin.

The above results are logical if one considers a void volume concept for the coating pigment (the volume between pigment particles). At low adhesive concentrations, the void volume for the pigment is relatively high. This permits the easy penetration of an oil or ink vehicle. As the adhesive concentration is increased, the void volume is decreased. This reduces the oil penetration. At the point of maximum printed gloss and maximum oil holdout, the adhesive concentration is sufficient to fill all the pigment voids with a network of binder molecules. This prevents the passage of large molecules, such as those of an oil. However, it does not mean that the coating is necessarily sealed to the passage of all molecules. It is quite logical to expect that there may be a sufficient void volume, because of the nature of the adhesive, to pass small molecules such as gases.

The paint industry would interpret the formulation that requires the least amount of adhesive for the maximum oil holdout as the critical pigment volume concentration or CPVC. The CPVC is that formulation where just enough adhesive is present to produce a dry continuous film with all the voids between pigment particles filled. This pigment volume concentration or PVC is expressed as the ratio of pigment volume solids to the total solids volume of the coating. The coating formulations in this report that show these above characteristics will be considered CPVC's. Below these CPVC's (excess of adhesive), the oil holdout and printed gloss values remain constant because the pigment voids are filled; and, therefore, no changes in oil absorption can take place.

The Adhesive Volume at the CPVC

The void volume resulting from the pigment packing, apart from the adhesive, should be the same for all coatings, since the pigment is unchanged. Therefore, the CPVC's for the three adhesive systems studied with this pigment should be similar. However, molecular shape, molecular structure, and adhesive orientation of the binders in the coatings are different, therefore, some variations in the CPVC's will exist. These variations are shown as follows: for 100 g. of predispersed, premium, domestic kaolin clay, the volume of dry adhesive needed to reach the CPVC is 22.2 ± 1.2 ml. for the acid-modified hydroxyethylated cornstarch, 25.9 ± 1.4 ml. for the soy protein, and 27.6 ± 1.2 ml. for the corn white dextrin. These values are based on the partial specific volumes for the adhesives. The volumes resulting from the soluble soap, calcium stearate, etc., were ignored because of the negligible amounts in the coatings. In using partial specific volumes for such calculations, the following assumption was made: the dry weight volume occupied by a gram of the adhesive in an infinitely dilute solution is the same as that occupied in the dry coating. Any variations from this are probably quite small.

The partial specific volumes for these adhesives were determined in this laboratory and are as follows: (1) acid modified hydroxyethylated cornstarch (2% hydroxyethyl ether by weight) in 0.1 *N* NaOH is 0.617 ± 0.002 ml. per g., (2) corn white dextrin in 0.1 *N* NaOH is 0.614 ± 0.002 ml. per g., (3) soy protein in 0.14% aqueous NH_3 is 0.720 ± 0.002 ml. per g.

The adhesive volume demands at the CPVC are very similar for the corn white dextrin and soy protein. The corn white dextrin and the soy protein are two different molecular species, and the results are not necessarily expected to agree, however, speculatively, both are considered to be somewhat globular in structure. In this respect, close agreement is reasonable. The adhesive volume demand for the acid-modified hydroxyethylated cornstarch is lower than that for the corn white dextrin. In this case, both are of the same basic molecular structure. The difference is probably the result of molecular shape and adhesive orientation variations. Linear molecules tend to become aligned in the coating. This would reduce the amount of adhesive as compared to a spherical polymer. In this respect, it is generally accepted that an acid modified cornstarch is somewhat linear as compared to other types of modification (*8*). This may explain in part, the lower CPVC for the acid-modified hydroxyethylated cornstarch. Secondly, the hydroxyethyl ether modification disrupts the symmetry of the molecular structure. This probably causes weaker associative bonds between adjacent molecules and would tend to make a better film former. This, too, may reduce the volume of adhesive needed to produce a continuous film.

Paper Gloss

Figures 1, 2, and 3 all show general decreases in the calendered paper glosses as the adhesive concentrations are increased. Minima are reached at or near the CPVC's. This general decrease is logical, if one considers paper gloss a function of the clay particle orientation in the coating. This property, paper gloss as a function of clay particle orientation, has been shown by Comer, Stetson, and Lyons, and Kraske (*9, 10*).

As the adhesive concentration is increased there is a general increase in the amount of adhesive-pigment bonding. Upon supercalendering, this will make any shifting or reorientation of pigment particles, necessary for better reflectance, more difficult. This can be interpreted by a decrease in gloss. At the CPVC a maximum amount of adhesive-pigment bonding will exist, since the voids between the pigment particles are now filled. This will account for the minimum gloss. Below the CPVC there is an excess of adhesive with no increase in the number of adhesive-pigment bonds. Under these conditions, the pigment particles are surrounded by adhesive molecules. Therefore, upon supercalendering, the action is primarily on the excess adhesive that surrounds the pigment particles. If this adhesive is somewhat plastic in the calender nip or if there is weaker bonding between adjacent adhesive particles than adhesive-pigment particles, it would be logical to expect an increase in paper gloss as was found for these adhesives.

Following the above line of thought, it was not surprising to see the hydroxyethylated starch coatings supercalender to higher glosses than those of the corn white dextrin. Here the hydroxyethyl ether modification has probably caused weaker associative bonds between the adjacent starch molecules. This would result in a more "plastic" material, thereby giving easier finishing.

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Determination of Optical Properties of Pulp

J. H. E. HERBST and H. KRÄSSIG

A method for the determination of relative absorption and scattering coefficients is presented. When pulp is dyed under suitable conditions, the plot of $(1 - R_\infty)^2/2R_\infty$ (R_∞ = reflectance of a thick sheet) against the amount of dye added is linear, in agreement with the Kubelka-Munk equation. The slope of this plot gives the relative scattering coefficient, and the relative absorption coefficient is calculated from the relative scattering coefficient and R_∞ for the undyed material. The following advantages, all due to the use of thick sheets, are claimed: simplicity of procedure, flexibility of the conditions under which the method may be used, and avoidance of errors which are a function of the basis weight. Information on several variables affecting the results is given.

THE optical properties of materials which partly absorb light, partly transmit it, and partly reflect it diffusely are characterized by the scattering and absorption coefficients. The scattering coefficient is a measure of interfacial area (*1–5*), while the absorption coefficient is a measure of colored material present (*6, 7*).

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Kubelka and Munk (*8*) derived equations relating the thickness of the sample, its reflectance, the reflectance of the background, and the scattering and absorption coefficients. Van den Akker (*9*) proposed that the basis weight, rather than the thickness, be used in the calculation. Thus, it is possible to calculate the two coefficients from two reflectance measurements, at least one of which is performed on a thin sheet placed on a background of known reflectance, and from the basis weight of the thin sheet. Reviews on this subject were published by Kortüm and Vogel (*10*) and Van den Akker (*11*).

The present paper describes a method for the determination of the two coefficients, employing only sheets of infinite basis weight. Such a method is desirable for the following reasons:

1. It is quick and convenient.
2. Loss of fines is reduced to a minimum. Judd (*12*) tentatively attributed inconsistencies in the experimental results to loss of fines.
3. It is sometimes difficult to make the desired type of thin sheet. Thus, trouble was experienced in the preparation of thin sheets from butanol suspension (*1, 2*).
4. The literature contains references to irregularities depending on the basis weight. Judd (*12*) found reflectivities and scattering coefficients to be slightly lower at low basis weights than at high basis weights. In contrast with this, Giertz (*13*) reported that, at basis weights of less than 35 g. per sq. m., high values of the scattering coefficient were obtained. Stenius (*14*) found that the measured value of the reflectance at infinite basis weight was somewhat high in relation to the measured values on thin sheets, and that the scattering coefficient decreased with decreasing basis weight. Stenius argued that this is due to a higher scattering coefficient in the center of the sheet than on the outside. We conclude that agreement is lacking on what causes deviations which are functions of the basis weight.

Since the scattering coefficient depends on the unbonded interfacial area, it is a characteristic of a sheet, and not of a pulp. Scattering coefficients may be used to compare pulps only if conditions affecting the interfacial area are considered. Two important factors affecting the interfacial area for a given pulp are the liquid medium from which the sheet is made (*1, 2*) and the wet pressure (*1*). In addition, the interfacial area is increased if the pulp is dried and rewetted (*1*).

THEORY

The method is based on the fact that dyeing changes the absorption coefficient without changing the scattering coefficient. Thus, the Kubelka-Munk equation for sheets of infinite basis weight is:

$$\frac{k}{s} = \frac{(1 - R_\infty)^2}{2 R_\infty} \qquad (1)$$

where

k = the absorption coefficient
s = scattering coefficient
R_∞ = reflectance of a sheet of infinite basis weight

This equation may be modified to apply to a dyed sheet:

$$\frac{k + K}{s} = \frac{(1 - R_\infty)^2}{2 R_\infty} \qquad (2)$$

where K = absorption coefficient of the dye.

Since

$$K = cw \qquad (3)$$

where

c = proportionality constant for a given dye
w = mg. dye per g. pulp

equation (2) is the equation of a straight line with y-intercept k/s and slope c/s, when $(1 - R_\infty)^2/2 R_\infty$ is plotted along the ordinate and w along the abscissa. The straight line is plotted, the relative scattering coefficient $s_R = s/c$ is calculated from the slope, and the relative absorption coefficient $k_R = k/c$ is calculated from R_∞ of the undyed sheet by equation (1). Thus, unless c is determined, the method gives relative results only.

EXPERIMENTAL

Choice of Dyes. A dye is suitable if equation (3) applies to pulp-dye systems in which it is present. Two dyes, kindly supplied by Canadian Industries, Ltd., were found to fulfill this requirement. They were CR Chlorazol Black B 200 (actually a blue dye) and Durazol Yellow 6GS. Under the conditions used, the amount of unsorbed dye was a small (1–2%) constant percentage of the amount of dye added.

As pointed out by Van den Akker (*9*), the color of a dye may be changed by sorption. Table I shows that this was the case. However, there was no noticeable shift in the absorption maximum in reflection over a wide range of w for either dye.

Table I. Absorption Maxima (Millimicrons)

	Transmission	*Reflection*
Blue dye	575	615
Yellow dye	385	415

Typical Results. Typical results for pulp 1 (*cf.* Table II) are shown in Figs. 1 and 2 (experimental methods are discussed in Appendix I). The different symbols represent experiments performed on different days. At low values of w, the linear relationship predicted by the theory was obtained. At high values of w, $(1 - R_\infty)^2/2 R_\infty$ was lower than predicted by theory, and there was more scatter. This is attributed, respectively, to incomplete sorption at high values of w and the fact that, at lower values of R_∞, $(1 - R_\infty)^2/2 R_\infty$ changes very rapidly with R_∞.

The coefficient of variation* of s_R was 0.8% for the yellow dye and 1.2% for the blue dye. Coefficients of variation calculated for other pulps showed that these were representative values, and that the coefficient of variation for the blue dye was always somewhat higher than that for the yellow dye. A further indication that the yellow dye satisfies equation (3) somewhat better than the blue dye is that the graph ceased to be linear at substantially lower values of w with the blue dye than with the yellow dye for all pulps tried.

Different Pulps; Effect of Dye. Relative scattering coefficients for five pulps are given in Table II.

If the pulps behaved similarly at the two wavelengths, s_R^{415}/s_R^{615} would be the same for all pulps.

* Coefficient of variation = (standard deviation/mean) × 100; the slope of the straight portion was calculated by the method of least squares.

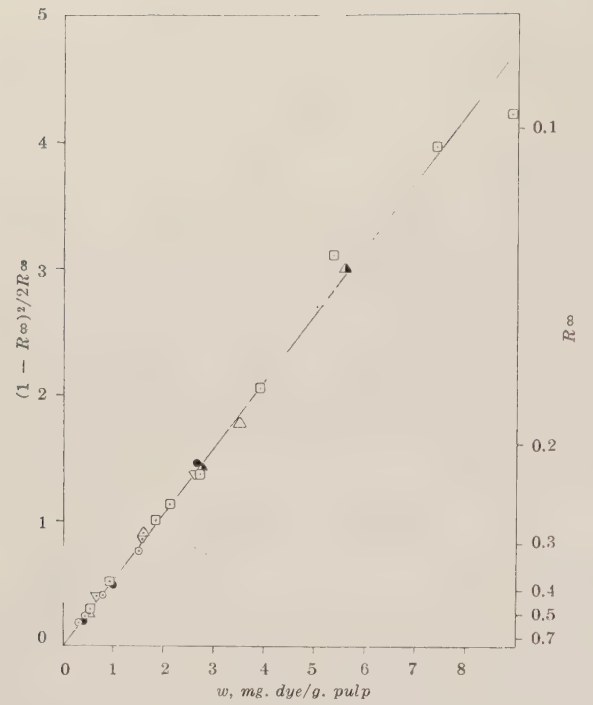

Fig. 1. Yellow dye

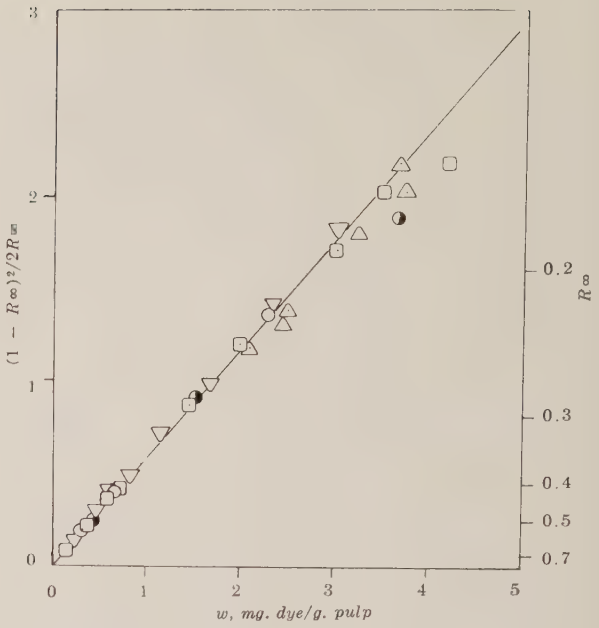

Fig. 2. Blue dye

As expected, this is not true. The difference in wavelength response appears to be roughly related to the severity of alkali treatment during processing.

We attempted to determine whether this difference is a dye effect or a wavelength effect by calculation of s_R^{615} for the yellow dye and of s_R^{415} for the blue dye. Sufficiently accurate results were not obtained because absorption was too feeble.

Table II. Different Pulps; Effect of Dye

Pulp No.	Pulp Description	s_R Yellow dye, 415 μ	s_R Blue dye, 615 μ	$\frac{s_R^{415}}{s_R^{615}}$
1	Softwood sulfite paper pulp	1.90	1.68	1.13
2	Hardwood sulfite dissolving pulp	2.65	2.23	1.19
3	Softwood kraft paper pulp	1.73	1.69	1.03
4	Softwood kraft paper pulp	2.00	1.84	1.08
5	Gum kraft dissolving pulp	1.84	1.91	0.96

Effect of Unbonded and Bonded Areas. s_R was determined for sheets made from butanol suspension, but otherwise under similar conditions. Results are shown in Table III.

Table III. Sheets Made from Butanol and Water

Pulp No.	Pulp Description	s_R, Blue dye[a] Butanol	Water	$\frac{s_K^{H_2O}}{s_R^{BuOH}}$
1	Softwood sulfite paper pulp	2.93	1.68	0.57
2	Hardwood sulfite dissolving pulp	3.17	2.23	0.70
3	Softwood kraft paper pulp	2.32	1.69	0.73
4	Softwood kraft paper pulp	2.43	1.84	0.76
5	Gum kraft dissolving pulp	2.41	1.91	0.79

[a] The yellow dye was soluble in acetone (*cf.* Appendix I).

For each pulp, the scattering coefficient of sheets made from butanol was substantially higher than that of sheets made from water. On the assumption that butanol sheets are unbonded and that the scattering coefficient is a linear function of the unbonded area (*1*, *2*), $s_R^{H_2O}/s_R^{BuOH}$ represents the fraction of the total area which is unbonded in sheets made from water. This fraction, too, is apparently related to differences in the severity of alkali treatment during processing.

Another method of changing the bonded area is by changing the wet pressure. Results obtained on the two samples showing extreme behavior in Table III are listed in Table IV.

Table IV. Effect of Wet Pressure

Pulp No.	Pulp Description	s_R^{415} Wet pressure, p.s.i. 0	500	1500
1	Softwood sulfite paper pulp	1.90	1.45	1.31
5	Gum kraft dissolving pulp	1.84	2.16	1.92

Pulp no. 1 gave the expected (*1*) decrease in scattering coefficient with increasing pressure, caused by the disappearance of surfaces by bonding. The data on pulp no. 5 show an increase in the scattering coefficient at moderate pressure, and then a decrease at high pressure. Thus, another effect causing an increase in surface with increasing pressure was superimposed on the normally predominant bonding effect which causes a decrease in surface. The unusual properties of pulp no. 5 will be further discussed below.

The unbonded area is also increased by drying and rewetting (*1*). Pertinent information for pulp no. 1 is given in Table V.

Table V. Drying and Rewetting

	s_R^{415}	s_R^{615}
Never dried	1.90	1.68
Dried at 50% R.H. and 25°C., and rewetted	2.40	2.36

Comparison with Standard Method. Table VI lists scattering coefficients obtained by the standard method (Appendix I), and other pertinent information.

Table VI. Comparison with Standard Method

No.	Pulp Description	s_S[a]	s_R^{415}	s_S/s_R^{415}	Apparent density, g./cc.	Alpha-cellulose, %	Rayon cellulose, %
1	Softwood sulfite paper pulp	400	1.90	24	0.47	88	91
2	Hardwood sulfite dissolving pulp	600	2.65	23	0.45	89	95
3	Softwood kraft paper pulp	370	1.73	21	0.50	86	89
4	Softwood kraft paper pulp	400	2.00	20	0.46	86	89
5	Gum kraft dissolving pulp	510	1.84	28	0.24	98	99

[a] Standard method based on basis weight (g./sq. m.).

If the methods were entirely equivalent, s_S/s_R^{415} would be constant. Differences are introduced by a slight difference in wavelength (414 vs. 458 mμ) and in the wet pressure applied (50 p.s.i. for s_S, none for s_R). We believe that the latter difference is responsible for the high value of s_S/s_R^{415} for pulp no. 5, because of its unusual response to pressure. The unusual value of the apparent density of the sheet and the exceptionally high alpha- and rayon cellulose (*15*) contents suggest that pulp no. 5 has unusually poor bonding properties because of its low hemicellulose content. This view also explains the results shown in Table IV.

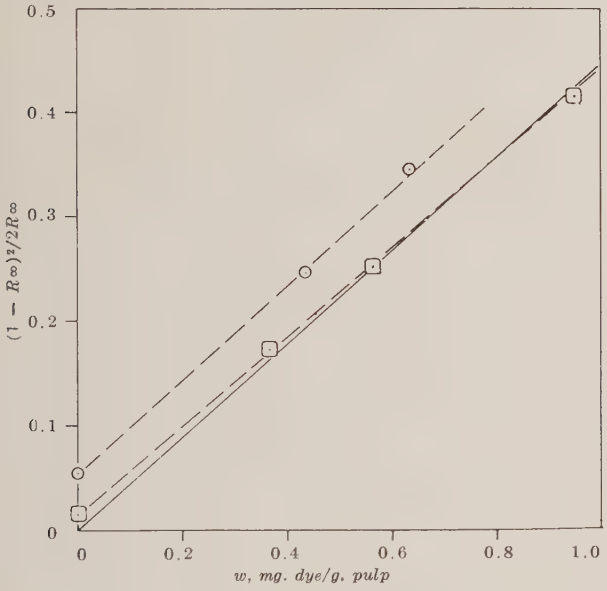

Fig. 3. Dark pulps

Circles, raw pulp; squares, chlorinated and alkali treated pulp; solid line, bleached pulp

Dark Pulps. The use of the method with dark pulps is illustrated in Fig. 3, showing graphs for a hardwood sulfite pulp in three stages of mill processing. The slopes are substantially the same, but the intercepts on the *y*-axis vary, being a function of the brightness of the undyed pulp.

Table VII. Brightnesses and Absorption Coefficients[a]

No.	Pulp Description	R_∞	$k_R \times 10^3$
1	Softwood sulfite paper pulp	0.931	2.7
2	Hardwood sulfite dissolving pulp	0.943	1.3
3	Softwood kraft paper pulp	0.886	8.5
4	Softwood kraft paper pulp	0.906	4.9
5	Gum kraft dissolving pulp	0.928	3.0

[a] Scattering coefficients for the yellow dye.

Absorption Coefficient and R_∞. Table VII lists R_∞ at 458 μ and values of k_R.

The results in Table VII suggest that, under some circumstances (small variations in s, high precision not required), R_∞ or $(1 - R_\infty)^2/2\ R_\infty$ is a satisfactory measure of the amount of coloring matter present; however, if these circumstances do not prevail, k is the only satisfactory parameter indicating the concentration of coloring matter.

Absorption Coefficient in Solution. Van den Akker (*9*) proved that, ideally, the absorption coefficient in solution, a, should be one half of k. In the present case, a/K should be the same for the two dyes. Actually, the ratio of a/K for the yellow dye to a/K for the blue dye was found to be roughly 0.7. This discrepancy is due, at least in part, to the wavelength shift shown in Table I.

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APPENDIX I

Experimental Methods

Preparation of Dyed Sheets. Ten grams (dry basis) of pulp which had never been dried, 25 g. of sodium chloride, the required volume of dye solution (1 g./l. blue dye, 0.125 g./l. yellow dye) and enough water to make the total volume 1 liter were shaken for 3 hr. The material was then formed into pads of 11 cm. diameter without washing and usually without applied pressure, and dried in air. R_∞ was measured on a General Electric recording spectrophotometer, usually at the wavelength at which maximum absorption occurred.

Tests were performed to determine the effect of additional stirring and the relative humidity at which the samples were dried. It was found that stirring for 15 min. with an electric stirrer after the shaking period had no effect on the results. The results were also found to be independent of the relative humidity, except at humidities higher than 90%.

For the preparation of sheets from butanol, the water was displaced from the dyed pulp by acetone, and the acetone by butanol (*1*).

Determination of s by Standard Method. Handsheets were prepared according to TAPPI Standard T 205 m, and their reflectance was measured against a black background. s was determined from this value, the basis weight, and R_∞.

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Lignin from the Alkaline Hydrogenation of Sugar Maplewood

III. Chromatographic Separation of Higher Molecular Weight Fractions and a Structural Study

ANIL BHATTACHARYA and CONRAD SCHUERCH

The ether-soluble phenolic fraction of lignin prepared by alkaline hydrogenation pulping has been separated by chromatography on alumina (activity grade IV) into a number of fractions, the first ones of which contain all known monomeric phenols from this preparation. Thereafter a series of higher molecular weight products are eluted. This chromatographic separation should be a convenient technique for studying variations in lignin products with changes in hydrogenation pulping conditions or wood species. The structure of one of the higher molecular weight fractions which was most cleanly separated from others and appeared reasonably homogeneous was investigated by a variety of methods. It is apparently dimeric and tentative structural conclusions are advanced. The periodate-permanganate reagent of Lemieux and Rudloff was used to destroy phenolic rings in this lignin fraction and leave side chain residues as substituted acids. This application of the reagent can probably be extended to related natural products with profit.

THE lignin produced from hardwoods by alkaline pulping in the presence of hydrogen under pressure and nickel catalysts is a comparatively attractive material as a source of phenols and as a substrate for studies on the structure of lignin. Previous studies (*1*, *2*, *3*, *4*, *7*), have shown that about one-fourth of the original lignin is present as three substances guaiacyl- and syringylethane and syringylethanol; a small percentage is present as the corresponding propanols; a small percentage is water-soluble and oxygen-labile and probably derived from pyrogallol and catechol, and a fraction of a per cent consists of six phenolic acids. These results have been obtained by means of fractional distillation, counter-current distribution, and for the acids, paper chromatography.

We have now developed a column chromatographic method for the separation of monomers and higher molecular weight material from the ether-soluble phenolic fraction of hydrogenated lignin. The method appears sufficiently reproducible to be used to study changes in lignin products with variations in the reaction conditions and wood source and to separate some individual compounds from the product mixture.

A variety of methods were tested without success before this was developed. Paper chromatographic methods which were successful for the separation of phenolic acids from this reaction were of little use with the phenolic fraction. Eight solvent systems were tested and of these six gave too high R_f values (over 0.8 for all monomers). In the other two cases the movement was very slow and there was little difference in the R_f values of the six monomers tested.

Reversed phase chromatography on paper and columns of treated cellulose, carbon, and celite proved unsuccessful in our hands. The solvents required to elute the phenols generally also extracted the stationary phase from the solid substrate. Untreated carbon columns irreversibly adsorbed phenols as Arlt (*1*) has previously shown and we were unable to decrease the activity of the carbon to a point where high recoveries could be obtained.

Chromatography on silicic acid was more satisfactory for the separation of monomers, but perhaps not entirely so for higher molecular weight species. In any case, minor changes in water content and operating conditions affected the rate of elution substantially and we therefore tested chromatography on alumina. Pepper and co-workers (*2*) have previously used a highly active form of alumina to separate various fractions of a lignin of this type from wheat straw and aspen. We have now found that by using alumina in a less active form, "Woelm" acid type containing 10% moisture (activity grade IV), the elution can be carried out with less polar solvents, the individual monomers are eluted at different rates (Table I), and higher molecular weight products are also reasonably well separated into a series of peaks (Fig. 1). It is probable that with minor changes in solvent schedule, the higher molecular weight products can be separated even more completely than is herein indicated.

This separation confirms the results of previous work in noting the great predominance of syringylethane (reported by Hibbert and co-workers (*2*, *3*, *4*) to correspond to at least 15% of the Klason lignin), the substantial amount of guaiacylethane (2% Klason lignin), and significant amounts of the corresponding ethanols and propanols. In addition it can be seen that there are still a few unidentified compounds with similar

Table I. Chromatography of Monomers on Alumina Columns

Monomer	*Peak effluent, vol.[a] ml.*
Guaiacylethane	70
Syringylethane	280
Syringylethanol	620
Guaiacylethanol	500
Syringylpropanol	630
Guaiacylpropanol	510

[a] Solvent schedule in text.

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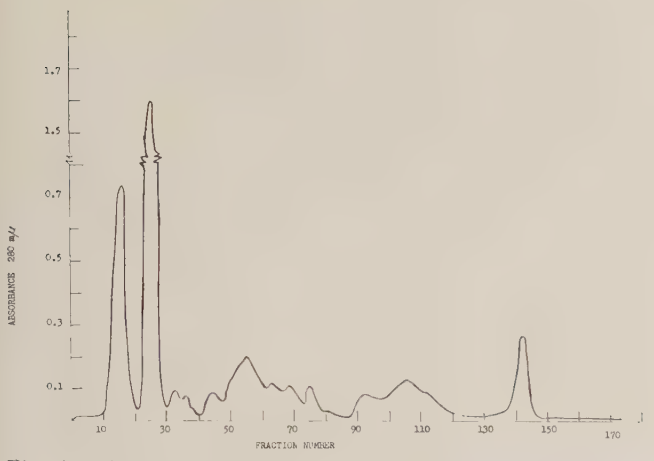

Fig. 1. Alumina column chromatography of lignin fraction F.B.$_1$

elution rates to the known monomers (peaks at fractions 33 and 36, for example) and a variety of substances with slower elution rates. On isolation the latter are powders or viscous greases and give every indication of being of higher molecular weight than the known monomers.

The fact that the peak at fraction 142 was substantially separate from the other products of hydrogenation, quite sharp and rather large, seemed to indicate that the product was reasonably homogeneous and was present in fair quantity. It was clearly not monomeric from its elution behavior, and on isolation proved to be an amorphous powder unlike the monomers which are oily or crystalline.

Partition chromatography of this fraction on cellulose columns impregnated with the aqueous phase of equilibrated butanol and phosphate buffer mixture and developed with the organic phase failed to separate the fraction into more than one peak. The partition coefficient of the material between the organic and aqueous phases was 1.24 and it is possible that a lower partition coefficient might have resulted in a separation which we were not able to achieve. By suitable modification of the chromatography on alumina, however, we were able to isolate this material in sufficient quantity for preliminary structural studies (about 1 g.).

A number of independent analyses showed good internal consistency. Thus the conductometric equivalent weight was 203. The systematic error on model compounds suggested that this value might be one to four per cent high.

The least possible molecular weight calculated from the methoxyl content of 16.25% was 191. The product therefore has one methoxyl group for each free phenolic hydroxyl and there is a free phenolic group for each aromatic ring.

The product was methylated to completion and the final methoxyl content was found to be 30.0% and the calculated molecular weight was 206 or that of the unmethylated product 192.

The Rast molecular weight on the methylated product was 433 which agreed with our presumption that the product was dimeric, calculated unmethylated: M.W. 405, but we failed to obtain consistent molecular weight data on the unmethylated product by cryoscopy in ethylene carbonate or camphor or by an isopiestic method in tetrahydrofuran. The values in these solvents, which we consider ureliable, were closer to that to be expected of a monomer. Confusing molecular weight data have been obtained previously on unmethylated tannins and lignin fractions. The carbon and hydrogen contents of the fraction were, respectively, 64.55% and 7.88%, and from these values an atomic ratio of $C_{3.13}H_{4.58}O_1$ is derived. The best molecular formula from this is $C_{22}H_{32}O_7$ which has a calculated molecular weight of 408, again in good agreement with that expected from the neutral equivalent, methoxyl, total hydroxyl, and molecular weight on the methylated product. However, the product has 2% of higher alkoxyl groups present as an impurity.

The ultraviolet spectrum of the fraction was typical of a phenolic material with a sharp minimum at 257 μ of K_{sp} = 4645 and a sharp maximum at 280 μ of K_{sp} = 8313. The difference spectrum between neutral and basic solutions of the fraction has the characteristic strong peak of an unconjugated phenol at 297 μ, K_{sp} = 10,024. Only a small rounded shoulder of flat peak was present near 360 μ K_{sp} = 2689. This indicated at most a small amount of conjugated phenolic impurity. Similarly the carbonyl region of the infrared spectrum had a diffuse and irregular band from 1675 cm.$^{-1}$ to 1800 cm.$^{-1}$ with a maximum at 1725, which was too weak to be considered an integral part of the structure but instead indicated some slight impurity. This is not surprising for the product was somewhat colored, perhaps from air oxidation. Other peaks were the C=O stretching frequency at 1450 cm.$^{-1}$, C=H stretching 2890 cm.$^{-1}$, OH stretching at 3588 cm.$^{-1}$, and phenyl ring at 3588 cm.$^{-1}$.

We also applied a method of degradation to this product which gave further data in general agreement with the preceding results. We investigated the application of the periodate-permanganate reagent of Lemieux and Rudloff (*5*) to the degradation of catechol, guaiacylethane, and our dimeric fraction 142. Periodate is known to cleave guaiacyl type aromatic nuclei yielding methanol in better than 90% yield and presumably derivatives of muconic acid (*6*). The trace of permanganate present hydroxylates double bonds to glycols, which are then cleaved to carbonyl groups and further oxidized by the permanganate to carboxyl groups. The permanganate is regenerated by periodate and the course of the oxidation can be followed quantitatively by iodometry.

As expected, catechol and 4-guaiacylethane consumed relatively rapidly 10 moles of periodate and the reaction reached an end point within 1 hr. This is consistent with the assumption that methoxyl cleavage requires about 1.5 moles and the two remaining double bonds require about 4 moles each. Fraction 142 consumed about 21 moles (assuming a molecular weight of 390), over a period of 2 hr. and there the reaction stopped. This is again consistent with the same amount of phenolic nuclei present as has been estimated from the methoxyl and phenolic hydroxyl content.

On extraction, the acidic products of this oxidation amounted to about 18% of the starting material and included a portion which after column and paper chromatography amounted to 3% of the starting material. This whitish jelly had a neutral equivalent of 106 and an infrared spectrum indicating only carboxylic acid bands.

The evidence so far collected indicates that fraction

142 is a relatively homogeneous material containing phenolic nuclei with equal numbers of free phenolic hydroxyl and methoxly groups. In theory this might be explained by the inclusion of *p*-hydroxyphenyl and syringyl units in equal numbers, but almost certainly indicates the presence only of guaiacyl units. The physical properties support the supposition that the material is dimeric and it is eluted from alumina, under conditions like those required by conidendrin. However, direct molecular weight determinations cannot at the present time be trusted. No significant amount of carbonyl or aliphatic hydroxyl groups are present nor can there be any conjugation α to the aromatic ring. Additional oxygen is present in the molecule and must be contained in ether linkages other than those of the benzyl type, which would be destroyed under the hydrogenation pulping conditions. The linkage between aromatic rings cannot be through the phenolic oxygen but presumably is through the side chains. The calculated molecule extinction coefficient in the ultraviolet is typical of analogous unconjugated phenols and indicates the absence of diphenyl or condensed type ring structures.

Beyond this, the structure of the aliphatic portion of this lignin fragment is seriously in doubt. Previous experience in other laboratories with lignans (Haworth) has shown that combustion data on material of this kind, especially when amorphous as in our case can be substantially in error because of solvent retention. If two guaiacyl nuclei are in fact subtracted from the calculated molecular formula of the fraction which we have obtained, the residual oxygens and hydrogens cannot be accommodated on the remaining carbon. In addition there is internal evidence that the fraction is not entirely pure and an unknown error is thus introduced.

In spite of these reservations, it appears that the preceding exploratory research makes a new series of lignin degradation products accessible to structural studies for the first time and gives some preliminary information on the structure of one of them. It is hoped that it will assist the application of classical organic methods to the problems of lignin structure and the course of chemical pulping which have been hampered by the limited number of useful degradations and inadequate methods of separation.

EXPERIMENTAL

Preparation of Hydrogenated Lignin

Sugar maple woodmeal (extractive free *Acer Saccharum*, Marshall, 40–80 mesh) was hydrogenated in an aqueous alkaline medium with Raney nickel and hydrogen, 750 p.s.i. starting pressure. Both a 2-liter Aminco rocking autoclave and a 2-gal. Aminco stirred autoclave were used. Conditions were as described previously (*7*) and the product was worked up in the usual way. The ether-soluble lignin amounted to 59% of the original Klason lignin and the chloroform solution remaining after bicarbonate extraction (*7*) was used in the following investigation.. This amounted to about 39% of original Klason lignin.

Small Scale Separation of Monomers and Ether-Soluble Bicarbonate-Insoluble Lignin

Ten grams of Woelm acidic alumina (grade I, made by M. Woelm-Eschwege, West Germany) was weighed in a wide-mouthed bottle and dried overnight in an electric oven at 105°C. After cooling in a desiccator, 1 ml. of water was added. The bottle was quickly stoppered, thoroughly sealed with tuck tape and allowed to stand overnight, or shaken mechanically for 3 hr. Enough cyclohexane was added to make a thin slurry which was poured into a chromatographic column (1.2 cm. diam.). A measured amount of a single monomer or a mixture of them (2.5 mg. of each in 0.5 ml. chloroform) was adsorbed on 0.5 g. of alumina, which after removal of solvent was transferred to the top of the column by cyclohexane. Similarly 30 mg. of the lignin fraction was placed on the column in the same way. The substrate was eluted by as much of the solvent schedule as was necessary: 110 ml. of cyclohexane followed by 110 ml. of cyclohexane chloroform mixtures of the following proportions: 90:10, 80:20, 70:30, 60:40, 50:50, 40:60, 30:70, 20:80, 10:90, and 220 ml. of pure chloroform and 95:5 and 90:10 chloroform butanol mixtures. All the solvent mixtures were saturated with water before use. One hundred-sixty fractions (10 ml. each) were collected.

Large-Scale Separation of Fraction 142

A concentrated solution of 50 g. of ether-soluble bicarbonate-insoluble fraction of hydrogenated lignin was adsorbed on the top of a column (5.4 cm. diam.) prepared out of 750 g. of Woelm acidic alumina containing 10% water. The solvent schedule was 8000 ml. of chloroform-cyclohexane (75:25), 8200 ml. of chloroform, 4500 ml. of 95:5 and 2500 ml. of 90:10 chloroform-butanol. All the solvent mixtures were saturated with water before use.

The chloroform-butanol extracts were combined and concentrated in vacuum. Care was taken to see that all the butanol was driven off. The residue was taken up in a minimum quantity of 75:25 chloroform-cyclohexane and adsorbed on the top of a 250-g. alumina column (2.2 cm. diam.) prepared in the usual manner. The column was eluted with 800 ml. of 75:25 chloroform-cyclohexane and 1000 ml. of pure chloroform. At this stage, adsorption of the eluate at 280 mμ was found to be nil. The column was next eluted with 300 ml. of 95:5, 300 ml. of 90:10, and 300 ml. of 80:20 chloroform-butanol mixtures. The fraction of 95:5 was concentrated in vacuum. The concentrated residue was dissolved in a small amount of chloroform and the solution was passed through a short carbon column. The solution was finally dried and evaporated in a vacuum oven at 40°C. The amorphous brownish residue weighing 0.98 g. was recovered.

Partition Chromatography of Fraction 142

Distribution coefficients of the fraction were determined between top and bottom phases of equilibrated butanol-1 and alkaline buffers. Butanol-1 and a phosphate buffer gave a K value of 1.24 whereas butanol-1 and a borate buffer gave a K value of 8.0. Partition chromatography was carried out with butanol-1 and phosphate buffer solvent system.

Butanol-1 was thoroughly equilibrated with phosphate buffer which was 9.4 molar in trisodium phosphate and 0.7 molar in tripotassium phosphate. The separated layers were stored in separate well-stoppered bottles. Six grams of dry cellulose powder was thor-

Table II. Ultraviolet Spectroscopy of Fraction 142

Medium	Wavelength, mμ		K_{sp}[a]	ΔK_{sp}[a]
	Maximum	Minimum		
95% aq. ethanol	280	257	8313	...
Aq. sodium hydroxide (pH 12)	292	277	13325	...
calcd.	297[b]	277[b]	...	10024

[a] Specific extinction coefficient in ml. per g. cm. at maximum of neutral, alkaline, or difference spectrum.
[b] Maximum and minimum of difference spectrum.

oughly shaken with the organic layer until a uniform dispersion was obtained. Aqueous layer was added dropwise with vigorous shaking until the dispersed cellulose just began to coagulate. A few more milligrams of cellulose powder were added with shaking until the dispersion became homogeneous again. The slurry was transferred to the chromatographic column (1.2 cm. diam.) with more of the organic layer. After the excess liquid had drained out of the column, 2.5 mg. of the compound was dissolved in a minimum quantity of the organic layer and the solution was put on the top of the column by a capillary. The column was eluted with 350 ml. of the organic phase. The emerging liquid from the column was passed through a differential refractometer. Ten-ml. fractions were collected. The absorbance of the fraction constituting a peak were determined at 280 mμ. Only one symmetrical peak was observed and the recovery of the material put on the column was 98%.

Analytical Results on Fraction 142

The sample was dissolved in dilute ammonia (4 *N*) and was titrated with 2.5 *N* lithium hydroxide conductometrically (*8*). An atmosphere of nitrogen was maintained in the cell and an ultramicroburet was used for titration. A number of monomeric and dimeric phenols were also titrated in the same way. Fraction 142 had a neutral equivalent of 203.

Methoxyl contents were determined by the method of Gran (*9*) and total alkoxyl by bromine titration. The methoxyl content was found to be 16.25% and the total alkoxyl as methoxyl 18.35%.

Carbon-hydrogen analysis was done by Spang Microanalytical Laboratory, Ann Arbor, Mich., and gave: C = 64.55%; H = 7.88%.

Ultraviolet spectra were determined in 95% aq. ethanol and aq. alkaline solution (pH 12) with a Cary recording spectrophotometer. The results are given in Table II.

Infrared spectra of the fraction, known monomeric and dimeric phenolic compounds were determined in a Baird Associates infrared spectrophotometer with a sodium chloride prism. Most of these were done in chloroform solution. The infrared spectrum of the acidic fraction of the products of periodate-permanganate degradation was determined in carbon tetrachloride solution. Some, α-conidendrin, for example, were ground with potassium bromide and made into pellets.

Methylation of Fraction 142

A weighed amount of the fraction (0.1132 g.) was dissolved in 20% aq. sodium hydroxide solution (15 ml.). The solution was stirred magnetically and an atmosphere of nitrogen was maintained over it during the reaction. The temperature was raised to 60° and dimethyl sulfate (3 ml.) was added dropwise during 1 hr. The solution was allowed to come to room temperature overnight, heated to boiling, cooled and acidified with dilute sulfuric acid. The acid solution was extracted with chloroform. The chloroform solution was washed with water until the latter was neutral to litmus. After drying over anhydrous magnesium sulfate, the solution was evaporated to constant weight in vacuum. The methoxyl content of the residue was determined in the usual way. The methylation operation had to be repeated three times before the methoxyl content of the product became constant. Owing to the insolubility of the product, the second and subsequent methylations had to be carried out in aqueous acetone medium. The final methoxyl content was found to be 30.05%.

Periodate-Permanganate Oxidation

Solutions of sodium metaperiodate (0.1 *M*) and potassium permanganate (0.1 *M*) were mixed so that the molar ratio between permanganate and periodate was approximately 1:10. The molar ratio of periodate to guaiacyl nucleus was roughly maintained at 20:1, and the final reaction mixture was so diluted that the concentration of periodate was below 0.1 molar.

In a typical rate study, the compound (0.132 mM.) was dissolved in 100 ml. water containing 11 ml. pyridine. This gave a pH of 8.6. The periodate-permanganate reagent was prepared by mixing 120 ml. of periodate solution (0.0936 *M*) and 10 ml. of permanganate (0.1 *M*) and diluting the mixture with water to 200 ml. At zero time, 5 ml. of each of the sample solutions and the reagent were mixed and kept in the dark. After the desired time interval, 10 ml. of saturated sodium bicarbonate solution, 10 ml. of sodium arsenite solution, and 2 ml. of 20% potassium iodide solution were added in that order and without delay. The mixture was allowed to stand for 10 min., a pinch of thyodene indicator added and the solution was titrated slowly with iodine solution to a stable blue end point. A blank determination with exactly the same amounts of reagents and after the same interval of time was also made in each case.

Recovery and Separation of the Products of Oxidation of Fraction 142

The sample (0.238 g.) was dissolved in 25 ml. pyridine in a glass stoppered bottle and diluted to about 200 ml. The solution was cooled in ice. The periodate-permanganate reagent (200 mM.) containing 23.8 mM. of periodate and 2.38 ml. of permanganate was added to the cooled solution and the reaction was allowed to proceed in the dark for 10 min. at 4°C. The cooling was discontinued and the reaction was allowed to proceed for a further period of 4 hr. at the end of which 2 g. of ethylene glycol was added to the mixture. After standing for 30 min., the pH of the solution was raised to 10. The precipitated manganese hydroxide was filtered off and the filtrate was acidified to pH 4 with hydrochloric acid. The resulting liquor was decolorized with sodium bisulfite and extracted exhaustively first with chloroform, then with ethyl acetate. The organic layers were dried with anhydrous magnesium sulfate, treated with a small amount of decolorizing carbon, filtered and concentrated in vacuum. The residue weighed 0.0431 g.

The oxidation products were dissolved in a minimum quantity of chloroform-methanol mixture (3:1) and the solution was applied as a streak on Whatman no. 1 paper (57 × 47 cm.). The paper was developed with butanol-1—1.5 *N* ammonium hydroxide (1:1, saturated organic layer) for 24 hr., dried, and cut into halves. The first half was extracted with chloroform methanol, the extract concentrated and further partitioned on a silicic acid column between organic and aqueous phases of benzene-methanol-water mixture (*10*) (10:7:3), then the eluate was titrated electrometrically. The fractions constituting the major peak were combined, acidified, and extracted with chloroform. The chloroform solution was washed free of mineral acid, dried, and concentrated in vacuum. A whitish jelly weighing 6.14 mg. was obtained.

The infrared spectrum of the jelly in carbon tetrachloride solution showed peaks at 2890 cm.$^{-1}$ and 1700 cm.$^{-1}$. The neutralization equivalent was found to be 106.

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Isolation and Properties of a Glucomannan from the Wood of White Birch (*Betula papyrifera*, Marsh.)

T. E. TIMELL

The larger part of the mannose residues in white birch wood has been found to originate from a glucomannan containing glucose and mannose residues in a ratio of 1:1.1. Partial hydrolysis of the polysaccharide gave 4-*O*-β-*D*-mannopyranosyl-D-mannose, 4-*O*-β-D-mannopyranosyl-D-glucose, 4-*O*-β-D-glucopyranosyl-D-mannose, and 4-*O*-β-D-glucopyranosyl-D-glucose. The methylated hemicellulose on hydrolysis yielded 2,3,6-tri-*O*-methyl-D-mannose, 2,3,6-tri-*O*-methyl-D-glucose, and tetra-*O*-methylglucose in a molar ratio of 32:27:1, while the number of hexose residues per average molecule was 60. The number- and weight-average degrees of polymerization of the nitrated polysaccharide were 69 and 120, respectively. It is concluded that the glucomannan is an essentially linear (1→4)-linked diheteroglycan containing approximately equal quantities of β-D-glucose and β-D-mannose residues. The hemicellulose is polymolecular and contains a minimum number of 70 hexose residues per average molecule. Problems encountered in isolation of glucomannans from wood are briefly discussed.

Glucomannans have been isolated in recent years from the wood of many gymnosperms, such as cedar, hemlock, pine, and spruce (*1*), where they constitute the main hemicellulose. The wood of the arborescent angiosperms contains only a few percentages of glucomannans and relatively little is presently known concerning their structural details. A glucomannan fraction has been isolated from the wood of trembling aspen (*Populus tremuloides*) containing (1 → 4)-linked mannose and glucose residues in a ratio of 2:1 (*2*). Similar polysaccharides with mannose and glucose in a ratio of 1.5:1 were somewhat later isolated from western red alder (*Alnus rubra*) and madrona (*Arbutus menzeii*) (*3*.)

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In previous investigations several polysaccharides were isolated from white birch (*Betula papyrifera*), including a cellulose (*4*, *5*), a 4-*O*-methylglucuronoxylan (*6*), an *O*-acetyl-4-*O*-methylglucurono-xyloglycan (*7*), and water-soluble polysaccharides from the wood (*8*), as well as a cellulose (*9*), a 4-*O*-methylglucuronoxylan (*10*), and a pectic acid (*11*) from the inner bark. In a preliminary communication (*12*) the isolation of a glucomannan from white birchwood was reported. The present paper is concerned with the constitution and molecular properties of this polysaccharide.

RESULTS

Earlier investigations have indicated that aqueous potassium hydroxide is a better solvent for wood xylans than sodium hydroxide (*13*), while the latter has a higher dissolving power for glucomannans (*14*). Addition of boric acid to aqueous potassium hydroxide, enhances its ability to extract glucomannans from wood (*15*). When a holocellulose from white birchwood was exhaustivlely extracted with potassium hydroxide, a pure 4-*O*-methylglucuronoxylan was recovered (*6*). The residual alpha-cellulose still contained all the mannose residues in the wood. Partial hydrolysis gave 4-*O*-β-D-glucopyranosyl-D-mannose, a disaccharide presumably derived from an occluded glucomannan (*16*).

These observations suggested that a suitable procedure for isolating a hardwood glucomannan should entail a preliminary delignification, followed by extraction with aqueous potassium hydroxide for removal of most of the xylan. It should then be possible to isolate a crude glucomannan by treating the residual material with aqueous sodium hydroxide containing

borate. Complexing with copper (*17*) or barium hydroxide (*18*) should finally result in a pure product.

White birchwood was accordingly delignified by either the chlorine (*19*) or the chlorite (*20*) procedures, and the resulting holocelluloses were exhaustively extracted with 24% potassium hydroxide. The 4-*O*-methylglucuronoxylans thus obtained contained no mannose residues. Subsequent extraction with 17.5% sodium hydroxide to which 4% boric acid had been added, gave crude glucomannans containing 13–18% xylose residues and glucose and mannose in a ratio of 1:1.1. Precipitations of the glucomannans from an alkaline solution with aqueous barium hydroxide (*18*) did not alter this ratio but lowered the xylose content to 3–5%. A summary of the results is presented in Table I. Precipitation with Fehling's solution (*17*) increased the mannose to glucose ratio to 1.3 to 1.5. The crude and purified glucomannans contained approximately 80% and 50–60%, respectively, of the original mannose residues in the wood.

Table I. Composition of Glucomannans

Method of isolation	*Mannose/glucose*	*Glucose/Xylose, %*
Chlorine Holocellulose		
Crude glucomannan	1.24	13
After three purifications	1.09	4
Chlorite Holocelluloses		
Crude glucomannan	1.05	14
After one purification	1.11	3
Crude glucomannan	1.05	18
After one purification	1.11	5

It is evident from these results that a relatively pure glucomannan can be isolated from the wood of white birch in high yield by the above sequence of extractions. The constant chemical composition of the various preparations indicates the presence of a true diheteropolymer and not, for example, a mixture of a glucan and a mannan. This conclusion was corroborated by results obtained on boundary electrophoresis of a glucomannan (*21*) when only one maximum was observed. The specific rotation of the hemicellulose, $[\alpha]_D - 35°$, was close to that reported for other hardwood glucomannans (*2*, *3*). When oxidized with periodate, the glucomannan consumed one mole of oxidant per anhydrohexose unit, thus suggesting the presence of a (1 → 4)-linked polysaccharide.

Treatment of wood with chlorine and ethanolamine is known to cause little or no depolymerization of polymers such as cellulose (*19*) or xylan (*22*, *23*) as long as some lignin still remains. Chlorous acid, albeit seemingly less drastic in its action, has repeatedly been shown to degrade polysaccharides quite noticeably (*19*, *22*, *24*, *25*). A glucomannan prepared from a chlorine holocellulose was accordingly used for molecular weight determinations. The polysaccharide was converted without any apparent degradation (*26*) into a nitrate. Osmotic pressure and light-scattering measurements with *n*-butyl acetate solutions of this derivative gave number- and weight-average molecular weights of 19,500 and 33,800, corresponding to degrees of polymerization of 69 ($\bar{P}_n$) and 120 ($\bar{P}_w$), respectively. The ratio $\bar{P}_w/\bar{P}_n$ was indicative of a Flory distribution of molecular weights.

The glucomannan was subjected to partial hydrolysis with aqueous formic acid (*27*) to give a mixture of sugars which was resolved on a charcoal-Celite column (*28*) by gradient elution with aqueous ethanol. Besides glucose and mannose, disaccharides and trisaccharides were obtained. The latter were not further examined. The disaccharides all crystallized and were identified as 4-*O*-β-D-mannopyranosyl-D-mannose (mannobiose), 4-*O*-β-D-glucopyranosyl-D-mannose, 4-*O*-β-D-mannopyranosyl-D-glucose, and 4-*O*-β-D-glucopyranosyl-D-glucose (cellobiose).

The isolation of the glucosyl mannose and the mannosyl glucose shows that glucose and mannose residues are chemically combined in the hemicellulose. The nature of the disaccharides indicates the presence of β-(1 → 4)-glycosidic bonds in the glucomannan. As usual, mannobiose was obtained in the highest, cellobiose in the lowest yield, and the amount of glucosyl mannose was ten times that of mannosyl glucose. Glucosyl mannose has previously (*16*) been isolated from white birchwood, and all oligosaccharides have recently been obtained on partial hydrolysis of a glucomannan (*29*) from the wood of red maple (*Acer rubrum*).

The glucomannan was converted to its fully substituted methyl derivative, a portion of which was subjected to methanolysis and then hydrolysis. The sugar mixture obtained was resolved on a charcoal-Celite column by gradient elution with aqueous ethanol. Three fractions were obtained, namely, 2,3,6-tri-*O*-methyl-D-glucose, which crystallized, 2,3,6-tri-*O*-methyl-D-mannose, identified through its characteristic 1,4-di-*p*-nitrobenzoate derivative, and finally a tetra-*O*-methylhexose. The latter was chromatographically identical with 2,3,4,6-tetra-*O*-methyl-D-glucose, and on demethylation gave glucose and a trace of mannose. The sugars were obtained in a ratio of 32:27:1, corresponding to one nonreducing end group per 60 hexose residues.

The number of hexose residues per average molecule was 71 as determined by osmotic pressure measurements. The glucomannan accordingly contained 1.2 nonreducing end groups per average molecule, corresponding to 0.2 branching points per molecule. This low value, in conjunction with the almost complete absence of any di-*O*-methylhexoses in the hydrolyzate from the methylated glucomannan, suggested that the polysaccharide was essentially linear. The isolation of the 2,3,6-tri-*O*-methylhexoses shows that the glucomannan was composed of (1 → 4)-linked hexose residues. The low specific rotations of the original and the methylated glucomannans indicate that the glycosidic linkages were of the β-type.

From this evidence a tentative structure can now be assigned to the glucomannan present in white birchwood. This hemicellulose is composed of approximately 70 β-D-glucopyranose and β-D-mannopyranose residues in an approximate ratio of 1:1.1. The hexose residues are linked together by (1 → 4)-glycosidic bonds to essentially linear macromolecules. Glucose constitutes the majority of the nonreducing end groups. The glucomannan is polymolecular and has a weight-average molecular weight approximately twice as high as the number-average value.

→4-β-D-Glc*p*-1→4-β-D-Man*p*-1→4-β-D-Man*p*-1→4-β-D-Glc*p*-1→

DISCUSSION

The present polysaccharide is very similar to the glucomannan previously isolated from the wood of red maple (*29*). Unlike the birch glucomannan, this hemicellulose has a ratio of glucose to mannose of 1:2, but otherwise there appear to be few differences in molecular structure. Both are linear, β-(1 → 4)-linked macromolecules and in both glucose forms most of the nonreducing end groups.

Recent studies on glucomannans from other arborescent angiosperms (*30*) suggest that most hardwood species contain glucomannans with a ratio of glucose to mannose of 1:1.5–2. The genus *Betula* apparently forms an exception in this respect since glucomannans from both the present species and from yellow birch (*Betula lutea*) contain almost equal amounts of glucose and mannose. Some birches are exceptional among the hardwoods also in their high xylan content.

Glucomannans from gymnosperms usually contain glucose and mannose residues in a ratio of 1:3 or 1:4. It should be emphasized, however, that the exact composition of wood glucomannans seems to be strongly dependent on the mode of isolation of the polysaccharides. Often, products obtained in low yields have a composition different from those isolated in high yields. Fractionation during isolation could easily occur because of differences in solubility properties (glucans are normally more soluble in aqueous alkali than mannans), or because of differences in location within the cell wall.

An example of this is furnished by the mannose-yielding portion of sitka spruce (*Picea sitchensis*). A glucomannan from this species has been reported (*31*) to contain only one glucose per 15 mannose residues, while in another investigation the ratio between glucose and mannose is stated to be 1:2.5 (*32*).

Glucomannans from Norway spruce (*Picea abies*) have been found (*33*) to contain a glucomannan, one fraction of which is easily extracted with alkali while another is more resistant. This behavior was tentatively explained in terms of differences in degree of polymerization, arrangement of the macromolecules, and location within the wood fiber. Similarly, during technical pulping processes, one portion of softwood glucomannans is often removed quite easily whereas the remainder is not eliminated even on prolonged cooking.

The present (crude) glucomannan contained the larger portion of the mannose residues present in the wood. The 20% not accounted for were probably also derived from a glucomannan, although the possibility that they originated from other polysaccharides cannot be excluded. When white birchwood was extracted with water a (*8*), mixture of polysaccharides was obtained which contained considerable amounts of mannose residues. The presence of a galactoglucomannan in similar water-soluble polysaccharides has been indicated (*34*).

EXPERIMENTAL

All specific rotations are equilibrium values and were determined at 20°C. in water at a concentration of 1.0 g. per 100 ml. unless otherwise stated. Melting points are corrected. Evaporations were carried out *in vacuo* at 40–50°C.

Paper Chromatography. Sugars were separated by the descending technique on Whatman no. 1 filter paper by the following solvent systems (v./v.): (A) ethyl acetate:acetic acid:water (9:2:2), (B) butan-1-ol:pyridine:water (10:3:3), and (C) butanone:water (89:11). Paper electrophoresis was carried out on Whatman no. 3MM filter paper in a 0.05 *M* borate buffer at 750 to 2,000 v. *o*-Aminodiphenyl was used as a spray reagent as well as for quantitative estimation of sugars (*35*).

Wood. A birch tree, evidently unaffected by birch dieback and approximately 70 years old, was cut in the spring of 1958, debarked, chipped, and converted to sawdust. The 40–60 and 60–80 mesh fractions were exhaustively extracted with ethanol-benzene (12:, v./v.) and subsequently with cold water. The extractive-free wood contained alpha-cellulose (47%), lignin (20.6%), pentosan (28.7%), and uronic anhydride (4.2%). The wood contained 1.8% mannose residues.

Isolation of Holocelluloses. Wood meal (185 g.) was treated three times with chlorine and alcoholic ethanolamine (*19*). The yield of chlorine holocellulose was 138 g. or 75% of the wood. Five similar treatments gave a total of 790 g. of chlorine holocellulose. For preparation of chlorite holocellulose, wood meal (1050) g. was suspended with stirring in 16 liters of water. Acetic acid (120 ml.) and sodium chlorite (360 g.) were added every hour for a total time of 8 hr. The chlorite liquors were removed by filtration and the residue was washed with water and then with ethanol. Yield: 775 g., corresponding to 74% of the wood.

Isolation of a Crude Glucomannan. Chlorine holocellulose (790 g.) was shaken with 24% (w./w.) aqueous potassium hydroxide (8 liters) at room temperature for 12 hr. The alkaline solution was recovered by filtration and added to four times its volume of ethanol containing an excess of acetic acid. The precipitate formed was washed on the filter with aqueous ethanol, ethanol, and petroleum ether to give a white powder (285 g.). Only xylose and uronic acids were obtained on hydrolysis.

The solid residue was exhaustively washed with water and then extracted with 17.5% sodium hydroxide to which 4% boric acid had been added (4 liters). The filtrate and washings on precipitation with ethanol and acetic acid gave a crude glucomannan (30.5 g., 2.9% of the wood) containing glucose (38%), mannose (47%), and xylose (15%) residues. Two products isolated from chlorite holocelluloses in a similar way contained 13.5 and 18% xylose residues and both had a ratio of mannose to glucose of 1.05.

Purification of the Glucomannan. Glucomannan from chlorine holocellulose (30 g.) was dissolved in 1.5 liters of 10% aqueous sodium hydroxide and 5% barium hydroxide (2.5 liters) was added (*18*). The precipitate formed was washed twice with 5% sodium hydroxide and three times with water and was then, after acidification with acetic acid, recovered in the usual way. Yield: 22 g., or 2.1% of the wood. Two additional treatments with barium hydroxide gave 7.0 g. of a glucomannan containing 4% xylose residues and a mannose-to-glucose ratio of 1.09. One similar treatment of the two products obtained from the chlorite holocellulose gave glucomannans containing 3 and

Table II. Osmometry Data for Nitrated Glucomannan

W [a]	h [b]	h/w
6.003	8.233	1.371
4.844	6.549	1.351
3.472	4.695	1.352
2.663	3.591	1.348
1.343	1.820	1.355
0	...	1.32

[a] Concentration in g./kg. solution.
[b] Osmotic height in cm. solution.

5% xylose, respectively, and with a mannose-to-glucose ratio of 1.11 in both cases.

Specific Rotation. The specific rotation of the glucomannan was determined by shaking a sample (150 mg.) with 10% sodium hydroxide (10–12 ml.) for 2 days. The solution was ultracentrifuged at 140,000 *g* for 30 min. and the clear solution was decanted and used for measurements. Two aliquots of 5 ml. each were precipitated into acid ethanol and the precipitate was recovered on an asbestos-covered coarse filter crucible. After washing with ethanol and petroleum ether the two crucibles were dried *in vacuo* at 60°C. and then weighed. The value obtained for $[\alpha]_D$ was −35° (*c*, 1.0 in 10% sodium hydroxide).

Periodate Oxidation. Glucomannan (150 mg.) was shaken for various lengths of time (20–230 hr.) with 0.05 *M* sodium metaperiodate (50.0 ml.). The consumption of oxidant was determined by the excess arsenite method. Extrapolation to zero time of the oxidation-time curve gave a consumption of periodate of 1.00 ± 0.02 mole per hexose residue.

Table III. Light-Scattering Data for Nitrated Glucomannan

Initial concn. × 10³ g./ml.	*Actual concn. × 10³, g./ml.*	$C/I_{90°}$
10.0	8.78	5.883
8.0	7.13	5.401
6.0	5.22	4.924
4.0	3.47	3.899
2.0	1.76	3.259
0	0	2.70

Osmotic Pressure Measurements. Glucomannan (3.0 g.) was treated with a mixture of nitric acid, phosphoric acid, and phosphorus pentoxide (64:26:10 w./w.) (*26*) at +4°C. for 2 hr. The reaction mixture was poured into ice-water and washed on the filter with ice-water until neutral. Drying *in vacuo* at room temperature over potassium hydroxide gave a grayish powder (4.8 g.). Nitrogen content: 13.28%, degree of substitution: 2.68, base molar weight: 283 (*36*).

Osmotic pressure measurements were carried out as described previously (*6*) with Stabin-Immergut (*37*) osmometers, gel cellophane membranes, and *n*-butyl acetate as a solvent. The temperature was 30°C. and the static method of measuring the osomotic height was employed. The results are presented in Table II. Extrapolation to zero concentration gave a value of 1.32 for $(h/w)_{w=0}$, corresponding to a molecular weight of 19,500 and a degree of polymerization of 69.

Light-Scattering Measurements. Light-scattering intensities were measured at 5460 A. with a Brice-Phoenix photometer of a modified design (*38*). The solvent for the glucomannan nitrate was *n*-butyl acetate. The solutions were clarified by ultracentrifugation at 140,000 *g* after which they were transferred to the light-scattering cells and subjected to centrifugation at 35,000 *g*. Scattering intensities were measured at 45°, 90°, and 135° relative to the incident beam at different concentrations. The results are given in Table III. The refractive index increment was determined with a Brice-Phoenix differential refractometer at 4358 A. and was 0.085 ml. per g. The molecular weight was calculated from the relationship $1/\bar{M}_w = H(C/\tau)_{C=0}$ where the symbols have their usual significance.

Partial Hydrolysis of the Glucomannan. Glucomannan (10.0 g.) was dissolved in 90% formic acid (50 ml.). Water (50 ml.) was added and the solution was heated in boiling water for 3 hr. (*27*). The solid residue was removed and again hydrolyzed in the same way. This treatment was repeated once. The solutions were combined and formic acid was removed by repeated evaporation with water. Formate esters were eliminated by boiling with 0.05 *N* sulfuric acid for 10 min. After neutralization with barium carbonate, evaporation gave a solid glass (8.0 g.). Paper chromatography (solvents A and B) indicated the presence of several oligosaccharides, glucose, mannose, and traces of xylose.

Resolution of the Partial Hydrolyzate. The hydrolyzate (7.5 g.) was added to the top of a column containing a 1:1 mixture of Darco G-60 charcoal and Celite (6 × 64 cm.). Gradient elution was carried out with 4 liters each of the following solvents: 2% aqueous ethanol → 15% ethanol, 15% ethanol → 30% ethanol, and 30% ethanol → 60% ethanol. Fractions, 25 ml. each, were collected at an average rate of one or two per hour. Every third fraction was examined by paper chromatography (solvent B). Experimental results are summarized in Table IV.

Table IV. Resolution of the Partial Hydrolyzate

Fraction no.	*Amount, g.*	*Component*
1–25	4.1	Glucose, mannose
45–90	0.80	Mannobiose
102–120	0.05	Mannosyl glucose
135–189	0.10	Oligosaccharide
204–216	0.05	Cellobiose
228–288	0.55	Glucosyl mannose
312–468	0.63	Trioses
630–720	0.30	Tetraose

Identification of Oligosaccharides. Only the four disaccharides were fully identified although several of the higher oligosaccharides crystallized. All disaccharides moved on the paper chromatogram at rates identical to those of authentic specimens. The sugars were crystallized twice from methanol containing water and butan-1-ol.

(*1*) *4-O-β-*D*-Mannopyranosyl-*D*-mannose.* This sugar had $[\alpha]_D$ −10° and m.p. and mixed m.p. 195 to 197°C. Hydrolysis gave only mannose.

(*2*) *4-O-β-*D*-Mannopyranosyl-*D*-glucose.* This disaccharide, which did not crystallize easily, had m.p. and mixed m.p. 198 to 199°C., $[\alpha]_D$ + 18°. Hydrolysis yielded equal amounts of mannose and glucose.

(*3*) *4-O-γ-β-*D*-Glucopyranosyl-*D*-glucose.* This sugar (cellobiose) had $[\alpha]_D$ +35°, m.p. and mixed m.p. 125 to 126°C. Hydrolysis gave only glucose.

(*4*) *4-O-β-*D*-Glucopyranosyl-*D*-mannose.* This disaccharide, which crystallized readily, had $[\alpha]_D$ +10°, m.p. and mixed m.p. 137 to 138°.

Methylation of the Glucomannan. Glucomannan (20 g.) was dissolved in 40% aqueous sodium hydroxide (350 ml.) in an atmosphere of nitrogen and dimethyl sulfate (300 ml.) was added dropwise over a period of 10 hr. This treatment was repeated seven times. The reaction mixture was neutralized with sulfuric acid. Most of the salts were removed by crystallization at +4°C. and the remaining solution was subjected to dialysis until salt-free. Freeze-drying gave 16.7 g. of material.

The partly methylated glucomannan was dissolved in dry dimethyl formamide (250 ml.) (*39*) and silver oxide (50 g.) and methyl iodide (50 ml.) were added. After shaking in the dark for 2 days at 30°C., the same amounts of reagents were again added. After an additional 2 days, the solid residue was extracted on the centrifuge with chloroform (3 liters). The chloroform was extracted once with 5% aqueous potassium cyanide and then twice with water, after which it was dried over anhydrous sodium sulfate and concentrated after filtration to 100 ml. The solution was poured slowly into petroleum ether (2 liters) to give a precipitate which was recovered by filtration, washed with petroleum ether and dried *in vacuo*. The infrared diagram of the product indicated the absence of hydroxyl groups.

Anal. OCH_3 = 44.0 %; $[\alpha]_D$ −30° (*c*, 1.0 in chloroform); yield: 9.0 g.

Hydrolysis of the Methylated Glucomannan and Resolution of the Hydrolyzate. The methylated glucomannan (6.0 g.) was boiled under reflux with 0.7 *N* methanolic hydrogen chloride (250) ml. for 6 hr. Most of the methanol was removed and the residue was refluxed with *N* hydrochloric acid for 7 hr. The solution was neutralized with silver carbonate and the sugar mixture (5.5 g.) was recovered in the usual way.

A portion of the hydrolyzate (1.9 g.) was added to the top of a column (3.5 × 55 cm.) containing Darco G-60 charcoal and Celite in a ratio of 1:1. Gradient elution was carried out with 4 liters each of 5% aqueous ethanol → 20% ethanol and 20% ethanol → 40% ethanol. Two tri-*O*-methylhexoses and one tetra-*O*-methylhexose were obtained in addition to traces of di-*O*-methylhexoses.

*Identification of 2,3,6-Tri-O-methyl-*D*-mannose.* This fraction (910 mg.), the first to be eluted from the column, gave mannose on demethylation with hydrobromic acid (*40*). The colorless sirup had $[\alpha]_D$ −6°. A portion (100 mg.) was converted to the characteristic 1,4-di-*p*-nitrobenzoate derivative (*41*), m.p. and mixed m.p. 187 to 188°C., $[\alpha]_D$ +33° (*c* 0.7 in chloroform) after three recrystallizations from ethyl acetate-petroleum ether (1:1).

Anal. Calcd. for $C_9H_{18}O_6$: OCH_3 = 41.9%. Found: OCH_3 = 40.0%. Calcd. for $C_{23}H_{24}O_{12}N_2$: OCH_3 = 17.9%. Found: OCH_3 = 18.1%.

*Identification of 2,3,6-Tri-O-methyl-*D*-glucose.* The colorless sirup (770 mg.) crystallized on standing, m.p. and mixed m.p. 119 to 120°C., $[\alpha]_D$ +69°, after three recrystallizations from ethyl ether.

Anal. Calcd. for $C_9H_{18}O_6$: OCH_3 = 41.9%. Found: OCH_3 = 41.4%.

Tetra-O-methylhexose. The last fraction (30 mg.) had a rate of movement on the paper chromatogram (solvent C) identical to that of 2,3,4,6-tetra-*O*-methyl-D-glucose or the corresponding mannose derivative. Demethylation with hydrobromic acid gave glucose together with a trace of mannose.

Molecular Weight of the Methylated Glucomannan. The number-average molecular weight of the methylated glucomannan was determined by osmotic pressure measurements at six different concentrations as described above. The solvent was a 9:1 mixture (v./v.) of chloroform and ethanol. The value obtained, 14,300, corresponded to a degree of polymerization of 71.

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Constitution of an Arabino-4-*O*-methylglucuronoxylan from the Wood of Eastern White Pine (*Pinus strobus*, L.)

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An arabino-4-*O*-methylglucuronoxylan has been isolated in a yield of 5% from the wood of eastern white pine. Partial hydrolysis of the wood gave D-galacturonic acid, 4-*O*-methyl-D-glucuronic acid, 2-*O*-(4-*O*-methyl-α-D-glucopyranosyluronic acid)-D-xylopyranose, a galacturonosyl glucose, and a galacturonosyl rhamnose. The methylated hemicellulose on hydrolysis yielded 2-*O*-methyl-D-xylose, 3-*O*-methyl-D-xylose, 2,3-di-*O*-methyl-D-xylose, 2,3,4-tri-*O*-methyl-D-xylose, 2,3,5-tri-*O*-methyl-D-arabinose, and 2,3,4-tri-*O*-methyl-D-glucuronic acid in a mole ratio of 8.6:12.3:50:1:5.0:10. After removal of the arabinose groups and methylation, hydrolysis gave mono-*O*-methyl-D-xylose, 2,3-di-*O*-methyl-D-xylose, and 2,3,4-tri-*O*-methyl-D-xylose in a ratio of 3.6:51:1. The number of xylose residues in the two methylated hemicelluloses were 123 and 94 per average molecule. Number- and weight-average degrees of polymerization of the original polysaccharide were 104 and 250. On the basis of this evidence it is concluded that the arabino-4-*O*-methylglucuronoxylan in white pine wood contains a slightly branched framework of at least 100 (1 → 4)-linked β-D-xylopyranose residues to which are directly attached terminal residues of (1 → 2)-linked 4-*O*-methyl-α-D-glucuronic acid and (1 → 3)-linked L-arabinofuranose, one acid being present per seven and one arabinose per nine xylose residues. The hemicellulose is polymolecular and in its original state probably contained an average of approximately 200 xylose residues.

Eastern white pine is the largest conifer in the northeastern part of this continent, where extensive areas were once covered with this stately tree. Excessive logging has since destroyed most of the original pine forests, but in later years, reforestation has again made this valuable lumber tree fairly widespread. The main polysaccharides of white pine wood are cellulose (43%), a glucomannan (14.6%), and an arabino-4-*O*-methylglucurono-xylan (7.2%). This paper is concerned with the constitution of the xylan, and especially with the mode of attachment of its arabinose groups and with the degree of branching and molecular size of the polysaccharide, properties which have not been unequivocally established for any of the few softwood xylans so far investigated.

Partial acid hydrolysis of the wood gave a mixture of sugars the acid portion of which was resolved on a column of anion exchange resin (*1*, *2*) to yield sugar acids which were purified by charcoal (*3*) or paper chromatography. The acids obtained included a hexuronic acid, a mono-*O*-methylhexuronic acid, and three aldobiouronic acids.

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The hexuronic acid was converted to D-galactose, identified through its 1-methyl-1-phenylhydrazone derivative, and was therefore D-galacturonic acid. Reduction of the methyl glycoside of the *O*-methylhexuronic acid with lithium aluminum hydride (*4*, *5*) gave 4-*O*-methyl-D-glucose, characterized as its osazone (*6*, *7*) and the acid was accordingly 4-*O*-methyl-D-glucuronic acid. Two of the aldobiouronic acids could be only partly characterized because of the paucity of material available. One was probably a galacturonosyl glucose and the other a galacturonosyl rhamnose. A 2-*O*-(D-galactopyranosyluronic acid)-L-rhamnopyranose has previously been obtained from the wood of maritime pine (*Pinus palustris*) (*8*), as well as from several other sources (*9–12*).

The methoxyl content and equivalent weight of the third aldobiouronic acid, $[\alpha]_D$ +84°, corresponded to that of an *O*-methylhexuronosyl pentose. Its infrared spectrum was identical to that of an authentic sample of 2-*O*-(4-*O*-methyl-α-D-glycopyranosyluronic acid)-D-xylopyranose (*13*). Reduction of the ester glycoside and hydrolysis gave 4-*O*-methyl-D-glucose and D-xylose. The aldobiouronic acid was converted to its ester glycoside, reduced and hydrolyzed to yield 3,4-di-*O*-methyl-D-xylose and 2,3,4-tri-*O*-methyl-D-glucose, both of which were identified through their aniline derivatives (*14*, *15*). Acetylation of the methyl ester methyl glycoside of the aldobiouronic acid gave the characteristic, crystalline methyl 2-*O*-[methyl(2,3-di-*O*-acetyl-4-*O*-methyl-α-D-glucopyranosyl)uronate]-3,4-di-*O*-acetyl-α,β-D-xylopyranoside (*16*, *17*). This evidence shows that the acid was 2-*O*-(4-*O*-methyl-α-D-glucopyranosyluronic acid)-D-xylopyranose.

While the origin of the first two aldobiouronic acids was unknown, the galacturonic acid was probably derived from the pectic acid known to be present in the wood of white pine (*18*). The two 4-*O*-methyl-substituted uronic acids were assumed to be associated with the xylan constituent of the wood.

For isolation of this hemicellulose, extractive-free wood meal was delignified with acid chlorite (*19*) to give a holocellulose which was extracted with aqueous potassium hydroxide. The mixture obtained contained approximately 5 parts of an acidic xylan and 3 parts of a glucomannan, in addition to minor quantities of other polysaccharides. The crude xylan was purified by the barium hydroxide method (*20*) which after five treatments gave a product containing only xylose, arabinose, and uronic acid residues. It is evident from Table I that the ratio of xylose to arabinose increased with each treatment with barium

Table I. Purification of the Crude Xylan by the Barium Hydroxide Method

	Number of treatments				
	0	*2*	*3*	*4*	*5*
Yield, %	15	11	7	5	5
Glucose and galactose, %	23	14	11	3	Nil
Mannose, %	14	4	Nil	Nil	Nil
Xylose/arabinose	2.1	4.9	6.4	8.0	9.0

hydroxide, thus indicating a gradual removal of arabinose-containing material. The yield of pure polysaccharide corresponded to 70% of the amount originally present in the wood.

The hemicellulose, $[\alpha]_D$ −65°, was easily soluble in water or alkali. Its methoxyl content, uronic anhydride content, and equivalent weight suggested the presence of seven xylose residues per each 4-*O*-methylglucuronic acid residue, and its content of arabinose corresponded to one arabinose per nine xylose residues. Boundary electrophoresis gave only one peak, thus suggesting that the polysaccharide was chemically homogeneous.

A portion of the hemicellulose was methylated ten times with dimethyl sulfate and alkali and subsequently with methyl iodide and silver oxide in the presence of dimethyl formamide (*21*). The infrared diagram and methoxyl content of the final product indicated complete substitution. The methylated hemicellulose, $[\alpha]_D$ −60°, was subjected to methanolysis and the mixture of glycosides was resolved into a neutral and an acid portion on an anion exchange resin. The acid fraction contained only one component, the ester glycoside of which was reduced to give the crystalline methyl 2-*O*-(2,3,4-tri-*O*-methyl-α-D-glucopyranosyl)-3-*O*-α,β-D-xylopyranoside (*16*, *22*), thus showing that the original acid was 2-*O*-(2,3,4-tri-*O*-methyl-α-D-glucopyranosyluronic acid)-3-*O*-methyl-D-xylopyranose.

The neutral glycosides were hydrolyzed to the corresponding mixture of neutral sugars which was resolved on a charcoal-Celite column (*23*) by gradient elution (*24*) with aqueous ethanol (*25*). Complete separation of each of the five compounds present was achieved in one operation and all sugars were crystallized and/or identified through crystalline derivatives. The amount of each sugar obtained, in conjunction with the quantity and constitution of the partly methylated aldobiouronic acid, gave the composition of the hydrolyzate presented in Table II.

The large quantity of 2,3-di-*O*-methyl-D-xylose obtained together with the high negative rotation of the original and of the methylated hemicelluloses, indicated that the polysaccharide contained a backbone of (1 → 4)-linked β-D-xylopyranose residues. The free aldobiouronic acid, after methylation, contained a methoxyl group at C_4 in the xylose moiety, whereas the partly methylated aldobiouronic acid formed on degradation of the methylated polysaccharide was unsubstituted in this position. This fact, in conjunction with the absence of any 3,4-di-*O*-methyl-D-xylose in the hydrolyzate from the methylated hemicellulose, indicated that the acid groups were attached *directly* to the (1 → 4)-linked xylan framework. The nature of the aldobiouronic acid showed that these acid side chains consisted of (1 → 2)-linked 4-*O*-methyl-α-D-glucuronic acid residues.

Table II. Methylated Sugars Obtained on Hydrolysis of the Methylated Hemicellulose

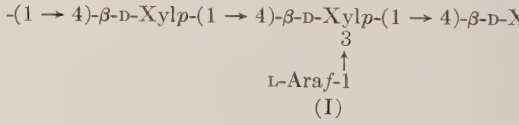
Component	*Moles*
2-*O*-Methyl-D-xylose	8.6
3-*O*-Methyl-D-xylose	12.3
2,3-Di-*O*-methyl-D-xylose	50.4
2,3,4-Tri-*O*-methyl-D-xylose	1
2,3,5-Tri-*O*-methyl-L-arabinose	5.0 (8.0)[a]
2,3,4-Tri-*O*-methyl-D-glucuronic acid	10.0

[a] Calculated from composition of original polysaccharide.

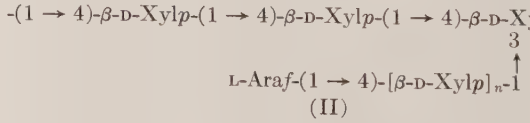
The molar composition of the hydrolyzate from the methylated hemicellulose indicated the presence of 71 xylose residues for each nonreducing group, the 2,3,4-tri-*O*-methyl-D-xylose being assumed to originate from the latter. When the number-average molecular weight of the methylated polysaccharide was determined by osmometry (*26*), a value of 25,500 was obtained, corresponding to 123 xylose residues in the xylan framework. The number of nonreducing end groups was accordingly 1.7, corresponding to 0.7 branch points per average molecule.

The isolation of 2,3,5 - tri - *O* - methyl - L - arabinose showed that the polysaccharide contained terminal residues of L-arabinofuranose. The amount of tri-*O*-methylarabinose obtained, 5.0 moles per mole of tri-*O*-methylxylose, was lower than the value expected from the composition of the original polysaccharide and the degree of polymerization of the methylated product, namely 8.0. This was probably due to preferential loss of the volatile 2,3,5-tri-*O*-methyl-L-arabinose during evaporations, a phenomenon which has been observed elsewhere (*22*, *27*). If this were so, the 8.6 moles of 2-*O*-methyl-D-xylose agreed well with the 8.0 moles of tri-*O*-methylarabinose, thus suggesting that the L-arabinofuranose residues were attached by (1 → 3)-glycosidic bonds to xylose residues. The excess of 0.6 mole of 2-*O*-methyl-D-xylose and 2.3 moles of 3-*O*-methyl-D-xylose were probably due to branching and to demethylation during the degradation of the methylated polysaccharide (*28*, *29*).

The above evidence does not establish whether the L-arabinofuranose residues were attached directly to the xylan backbone as in (I) or were linked through interposed xylose residues as in (II) (*30*). While two methods have supplied direct proof of the mode of



(I)

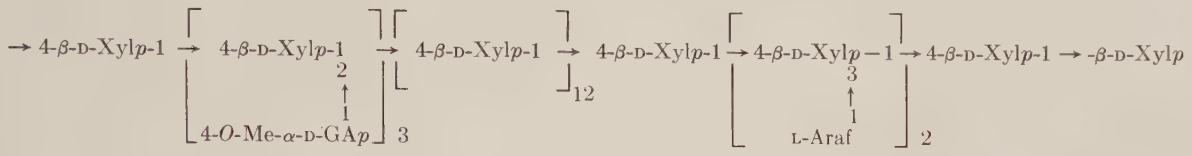


(II)

linkage of some of the arabinose groups in arabinoxylans (*31–33*), neither of them accounted for all arabinose residues involved. Another technique entailed a partial removal of the acid-labile L-arabinofuranose groups by mild hydrolysis (*34*), followed by estimation of the nonreducing end groups as compared to the number of such groups present in the original polysaccharide. Depolymerization of the xylan framework, even under conditions of mild hydrolysis, is impossible to avoid, however, and this causes an increase in the

number of nonreducing end groups. This method also fails to account for all arabinose groups present.

In an attempt to develop a better procedure, the hemicellulose was subjected to a mild acid hydrolysis which completely removed the arabinofuranose groups. Since each indirectly linked arabinose residue on removal gives rise to an additional nonreducing end group, a comparison between the number of such end groups present *per average molecule* in the original and in the hydrolyzed polysaccharide, would immediately establish how many, if any, of the arabinose groups that were so attached.



The arabinose-free hemicellulose was accordingly methylated to completion, a portion was subjected to methanolysis and the neutral glycosides were separated from the acid part. After hydrolysis, the neutral sugar mixture was resolved on a charcoal-Celite column by gradient elution to give a mixture of 2-*O*- and 3-*O*-methyl-D-xylose, 2,3-di-*O*-methyl-D-xylose, and 2,3,4-tri-*O*-methyl-D-xylose in a molar ratio of 3.6 : 51 : 1. If the xylose residue present in the partly methylated aldobiouronic acid is also taken into account, this corresponds to one nonreducing xylose end group per 64 xylose residues.

Osmotic pressure measurements indicated that the methylated hemicellulose contained 94 xylose residues, corresponding to 1.5 nonreducing end groups per average molecule. The corresponding value for the original polysaccharide was 1.7, thus indicating the same extent of branching. *All* arabinose groups were therefore attached directly to the xylan backbone as single, terminal side chains. If all of them had been indirectly linked, 11.7 nonreducing end groups would have been expected per average molecule of the arabinose-free polysaccharide.

Periodate oxidation of the hemicellulose indicated a consumption of 20.5 moles of oxidant per repeating unit of the polysaccharide. The calculated amount was 20.0 moles, thus suggesting the presence of an essentially (1 → 4)-linked polysaccharide.

Two different preparations of the hemicellulose were acetylated (*26*, *35*) and the number-average molecular weights of the acetates were determined from osmotic pressure measurements (*26*). The values obtained, 27,100 and 27,700, corresponded to degrees of polymerization (D.P.) of 101 and 104, respectively. The weight-average molecular weight was determined from light scattering measurements with dimethyl sulfoxide solutions of the polysaccharide (*36*). The value found was 43,100, corresponding to a D.P. of 250.

From the above evidence it is now possible to suggest a tentative structure for the arabino-4-*O*-methylglucurono-xylan present in the wood of white pine. This polysaccharide contains an average number of at least 100 β-D-xylopyranose residues linked together by (1 → 4)-glycosidic bonds to a framework containing slightly less than one branching point per average molecule. A single, terminal side chain of 4-*O*-methyl-β-D-glucuronic acid is directly attached by a (1 → 2)-linkage to every seventh, on the average, of the xylose residues. An L-arabinofuranose residue is directly linked by a (1 → 3)-glycosidic bond to every ninth of the xylose residues. Results obtained with other wood xylans (*37–40*) make it probable that the native polysaccharide originally contained also *O*-acetyl groups. The ratio between the weight- and number-average molecular weights (2.5) shows that the hemicellulose is polymolecular and possesses a Flory distribution (*41*) of molecular weights.

The present hemicellulose is similar, albeit not identical, to the acidic xylans previously isolated from other coniferous woods. The number of acid side chains, all of them 4-*O*-methylglucuronic acid residues, present in the softwood xylans so far studied varies between 1 per 4 and 1 per 7 xylose residues. Softwood xylans accordingly contain more acid groups than the 4-*O*-methylglucurono-xylans present in hardwoods, which normally appear to contain one acid group per 10 xylose residues. The only exception so far encountered in this respect seems to be the 4-*O*-methylglucurono-xylan present in white elm wood (*2*) which is as acidic as the softwood xylans (*42*).

The number of L-arabinofuranose residues present in the softwood xylans hitherto investigated exhibits greater variations. Xylans isolated from Norway spruce (*Picea abies*) (*43*) and loblolly pine (*Pinus taèda*) (*44*, *45*) are reported to contain no arabinose groups, but in another study (*46*) a xylan from the former species is stated to contain one arabinose side chain per six xylose residues. Xylans from Sitka spruce (*Picea sitchensis*) (*47*) and European larch (*Larix decidua*) (*48*) have been found to possess very few arabinose groups. Similar xylans from other conifers, however, undoubtedly contain arabinose side chains, with 12 xylose residues per arabinose for western hemlock (*Tsuga heterophylla*) (*22*), 10 for maritime pine (*Pinus palustris*) (*49*, *50*) and seven for Engelmann spruce (*Picea engelmanni*) (*51*).

It is not impossible that some softwood xylans are really devoid of arabinose residues or that some coniferous woods contain both an arabinoglucurono-xylan and a glucuronoxylan. The possibility, however, that some or all of the arabinose groups had been removed during the isolation of the first four of the above polysaccharides cannot be excluded. Such a loss could conceivably occur under the action of even mild acid hydrolysis or by removal of arabinose-rich portions of the hemicellulose during its isolation or purification, especially since the presence of arabinose side chains is known to increase the solubility of xylans (*22*). With the single exception of an aspen xylan (*52*), 4-*O*-methylglucuronoxylans from hardwoods appear to contain no arabinose groups (*42*).

In the present hemicellulose all arabinose side chains were attached directly to the xylan backbone. In this respect, the polysaccharide was thus similar to

arabinoxylans obtained from wheat-flour (*53*), rye-flour (*54*), barley-flour (*34*), and barley-husk (*55*). Whether the (1 → 3)-glycosidic bonds were of the α- or β-type cannot be decided on the basis of the present evidence, nor has this been established for any other arabinoxylan, although the occurrence of a β-linkage has been suggested for an arabino-4-*O*-methylglucuronoxylan from southern pine (*56*).

4-*O*-Methylglucuronoxylans isolated from Norway spruce (*43*) and European larch (*48*) are apparently linear, whereas a xylan from loblolly pine (*45*) probably contained two branching points per average molecule. The present polysaccharide was only slightly branched with respect to the xylan framework. Linear 4-*O*-methylglucuronoxylans of the same average molecular weight as the present hemicellulose are not soluble in water (*57*). The solubility in water of the present arabino-4-*O*-methylglucuronoxylan was probably due partly to its branched nature but mostly to its higher proportion of acid and neutral side chains (*22*). It is known that introduction of only a few *O*-acetyl groups in a 4-*O*-methylglucuronoxylan is sufficient to render the polysaccharide soluble in water (*37*).

Previous data indicate that softwood xylans probably contain a minimum of 100 xylose residues per average molecule (*43*, *48*). The present results lead to the same conclusion. It is well known, however, that polysaccharides suffer severe depolymerization under the action of the acid chlorite used here for preparing the holocellulose (*58*, *59*). In an earlier investigation (*57*) it was found that, under the delignification conditions used here, a 4-*O*-methylglucuronoxylan was degraded from an initial D.P. of 215 to a final value of 120. The number- and weight-average degrees of polymerization of the native arabino-4-*O*-methylglucuronoxylan in white pine wood might therefore have been in the neighborhood of 180 and 450, respectively. These are values almost identical to those found previously (*36*) for native 4-*O*-methylglucuronoxylans from various hardwoods. There is undoubtedly a considerable need for new delignification methods entailing no cleavage of glycosidic bonds. The modified chlorine-ethanolamine method (*58*) is useful for hardwoods, but it apparently fails for the more heavily lignified softwoods (*60*).

EXPERIMENTAL

All specific rotations are equilibrium values and were determined at 20°C. Melting points are corrected. Evaporations were carried out at 40 to 50°C. *in vacuo*.

Paper Chromatography

Solvents (v/v) used for separating the sugars were (a) ethyl acetate : acetic acid : water (9 : 2 : 2), (b) butan-1-ol : pyridine : water (10 : 3 : 3), (c) ethyl acetate : acetic acid : formic acid : water (18 : 3 : 1 : 4), (d) butanone-water (89 : 11), and (e) butan-1-ol : ethanol : water (40 : 11 : 19). Separations were carried out on Whatman no. 1 or (for preparative purposes) no. 3MM filter paper. Paper electrophoresis was carried out with the latter filter paper in a 0.05 *M* borate buffer (pH 9.4) and at 750 v. *o*-Aminodiphenyl was used as a spray reagent (*61*).

Composition of the Wood

Several healthy specimens of white pine, from southern Quebec, all 20 to 25 years old, were cut in the spring and fall of 1957. The wood was debarked, chipped, and converted to sawdust and the 20–80 mesh fractions were collected. The wood meal was exhaustively extracted with ethanol : benzene (1 : 2 v/v). Analyses by standard methods (*62*) gave the following composition of the wood: alpha-cellulose (43%), lignin (29.3%), pentosan (9.8%), acetyl (*40*) (1.2%), ash (0.2%), uronic anhydride (*63*) (4.0%), residues of glucose (46.0%) (*61*, *64*), mannose (11.7%), galactose (1.4%), arabinose (2.0%), and xylose (4.5%).

Isolation of Uronic Acids

Wood meal (100 g., 40–60 mesh) was treated at 30°C. with 72% sulfuric acid (170 ml.) for 1 hr. The mixture was diluted to 5 liters and boiled under reflux for 6 hr. After neutralization with barium hydroxide and filtration through Celite, the solution was treated with Amberlite IR-120 exchange resin concentrated and added to the top of a column with Dowex 1-X4 exchange resin (100 mesh, acetate cycle). Neutral sugars were removed by washing with water (negative Molisch test) after which acidic sugars were recovered by eluting with 3 *N* aqueous acetic acid (5 liters). Evaporation of the eluate yielded a syrup (2.5 g.). This process was repeated once.

Separation of Uronic Acids

The mixture of uronic acids (5.0 g.) was added to the top of a column (6 × 180 cm.) of Dowex 1-X4 exchange resin (100 mesh, acetate form). After washing with water (500 ml.), elution was carried out with aqueous acetic acid, increased in steps of 0.25 *N* from 0.25 *N* to 3.0 *N* with 2 liters of acid at each concentration. Fractions, 20 ml. each, were collected at a rate of 8 per hr. Five main fractions were obtained (I–V). Of these, fraction IV was a mixture of two higher uronic acids, presumably triouronic. These were not examined further.

Resolution of Fraction I

The first fraction to be eluted from the column (I, 250 mg.) on hydrolysis yielded galacturonic acid, glucose, and rhamnose, as indicated by paper chromatography (solvents A and B). Two somewhat overlapping spots were noted on the paper chromatogram with solvent C. Separation of a portion of the two acids by this technique yielded chromatographically pure sugar acids (20 mg. each).

Preliminary Characterization of Galacturonosyl Rhamnose and Galacturonosyl Glucose. The slower-moving component of fraction I on hydrolysis gave galacturonic acid and rhamnose and had $[\alpha]_D$ +8°C. (*c*, 1.0 in water). A portion of the acid (10 mg.) was converted to its methyl ester methyl glycoside by refluxing for 7 hr. with 0.7 *N* methanolic hydrogen chloride, reduced with lithium aluminum hydride (*4*, *5*) in tetrahydrofuran, and hydrolyzed with *N* sulfuric acid to give galactose and rhamnose. The second component gave galacturonic acid and glucose on hydrolysis and, after a similar reduction, galactose and glucose.

Resolution of Fraction II

The main portion of this fraction was an aldobiouronic acid but a monouronic acid was also present.

The mixture was added to the top of a cocoanut charcoal (*3*) column (4 × 35 cm.). Aqueous ethanol, 6 to 9%, eluted pure galacturonic acid (0.36 g.) and 10 to 14% ethanol gave a pure aldobiouronic acid (1.10 g.). Since the total amount added was 2.25 g., the recovery was not very satisfactory and use of this charcoal is not recommended.

Anal. Calcd. for $C_6H_{10}O_5$: Equiv. wt., 194. Found: Equiv. wt., 208; $[\alpha]_D$ +75°C. (*c*, 1.5 in water). Calcd. for $C_{12}H_{20}O_{11}$: OCH_3, 9.1%; equiv. wt. 340. Found: OCH_3, 9.3%; equiv. wt. 338; $[\alpha]_D$ +84°C. (*c*, 2.0 in water).

Identification of D-*Galacturonic Acid.* The mono-uronic acid (100 mg.) was boiled under reflux with 0.7 *N* methanolic hydrogen chloride for 6 hr. After neutralization with silver carbonate, filtration through Celite, treatment with hydrogen sulfide, filtration, and evaporation yielded a sirup (110 mg.) which was dissolved in dry tetrahydrofuran (60 ml.) and reduced with lithium aluminum hydride for 2 hr. at 40°C. The product was recovered and hydrolyzed in the usual way to give a sirup (60 mg.), $[\alpha]_D$ +90°C. (*c*, 1.5 in water), chromatographically indistinguishable from galactose (solvent B). The 1-methyl-1-phenylhydrazine derivative had m.p. and mixed m.p. 185 to 186°C.

Reduction and Hydrolysis of the Aldobiouronic Acid and Identification of the Hydrolysis Products. The aldobiouronic acid obtained from the charcoal column (630 mg.) was converted to its methyl ester methyl glycoside and reduced in the usual way with lithium aluminum hydride (2 g.) in dry tetrahydrofuran (75 ml.). The disaccharide (500 mg.) was hydrolyzed by refluxing for 6 hr. with *N* sulfuric acid to give a sirup (380 mg.). Two spots of equal intensity were obtained on the paper chromatogram (solvent D), corresponding to xylose and 4-*O*-methylglucose. The mixture was resolved on filter paper (solvent E) to yield D-xylose (86 mg.), m.p. and mixed m.p. 142 to 143°C., $[\alpha]_D$ +19°C. (*c*, 1.0 in water), and 4-*O*-methyl-D-glucose, $[\alpha]_D$ +60°C. (*c*, 2.5 in water) (*6*, *7*).

Anal. Calcd. for $C_7H_{14}O_6$: OCH_3, 16.0%. Found: OCH_3, 15.7%.

Recrystallization of the 4-*O*-methyl-D-glucosazone from aqueous ethanol gave yellow crystals, m.p. and mixed m.p. 155 to 156°C.

Methylation, Reduction, and Hydrolysis of the Aldobiouronic Acid. The ester glycoside of the aldobiouronic acid (520 mg.) was dissolved in dry dimethylformamide (12 ml.) and treated in the dark with silver oxide (10 g.) and methyl iodide (10 ml.) (*21*). After 48 hr. at room temperature, the mixture was extracted with chloroform and the recovered disaccharide was subjected twice more to the same treatment. After the third methylation, the chloroform extract was washed once with 5% aqueous potassium cyanide (*21*) and then twice with water (50 ml. each). The sirup obtained on evaporation (400 mg.) was dissolved in methyl iodide (10 ml.) and shaken with silver oxide (5 g.) at room temperature for 24 hr. The infrared diagram of the sirup (386 mg.) indicated the absence of hydroxyl groups and was identical to that of an authentic sample (*13*) of methyl 2-*O*-[methyl(2,-3,4-tri-*O*-methyl-α-D-glucopyranosyl)uronate]-3,4-di-*O*-methyl-D-xylopyranoside.

Anal. Calcd. for $C_{18}H_{32}O_{11}$: OCH_3, 51.2%. Found: OCH_3, 51.2%.

The fully methylated aldobiouronic acid (365 mg.) was reduced with lithium aluminum hydride (1.2 g.) in dry ethyl ether (85 ml.) to yield a sirup (300 mg.) which was boiled under reflux with 2 *N* methanolic hydrogen chloride (20 ml.) for 10 hr. and subsequently hydrolyzed by refluxing with *N* hydrochloric acid (30 ml.) for 8 hr. The sirup obtained (248 mg.) was resolved on the paper chromatogram (solvent D) to give a di-*O*-methylxylose (90 mg.) and a tri-*O*-methylglucose (71 mg.). Both sugars were purified by treatment with Amberlite IR-120 exchange resin and Darco G-60 charcoal.

*Identification of 3,4-Di-O-methyl-*D-*xylose and 2,3,4-tri-O-methyl-*D-*glucose.* The rate of movement of the di-*O*-methylxylose on the paper electrophoretogram was identical to that of an authentic sample of 3,4-di-*O*-methyl-D-xylose. The 3,4-di-*O*-methyl-*N*-phenyl-D-xylosylamine had m.p. 114 to 116°C. (*14*).

Anal. Calcd. for $C_7H_{14}O_5$: OCH_3, 34.8%. Found: OCH_3, 34.6%; $[\alpha]_D$ +25°C. (*c*, 1.0 in water).

*Preparation and Identification of Methyl 2-O-[methyl-(2,3-di-O-acetyl-4-O-methyl-α-*D-*glucopyranosyl)uronate]-3,4-di-O-acetyl-*D-*xylopyranoside.* The methyl ester methyl glycoside of the aldobiouronic acid (250 mg.) was dissolved in pyridine (75 ml.) and treated with acetic anhydride (25 ml.) at 25°C. for 20 hr. The solution was poured into water (150 ml.) and the acetate was recovered as described previously (*16*, *17*). The sirup (250 mg.) was dissolved in boiling ethyl ether. The crystals formed (60 mg.), on recrystallization from ethanol : ether (1 : 1) had m.p. and mixed m.p. 199 to 200°C. Their x-ray and infrared diagrams were identical with those of an authentic specimen (*17*).

Anal. Calcd. for $C_{22}H_{32}O_{15}$: OCH_3, 17.3%. Found: OCH_3 17.0%; $[\alpha]_D$ +95°C. (*c*, 1.1 in chloroform).

Resolution of Fraction III

This fraction was resolved on the paper chromatogram (solvent A) to yield higher uronic acids, which were not further examined, and D-galacturonic acid, identified as described above by conversion to D-galactose.

Identification of 4-O-Methyl-*D-*glucuronic Acid (Fraction V)

The mono-*O*-methylhexuronic acid was converted to its methyl ester methyl glycoside (100 mg.) and reduced with lithium aluminum hydride in tetrahydrofuran. The reduced product (60 mg.) was chromatographically indistinguishable from 4-*O*-methylglucose. The 4-*O*-methyl-D-glucosazone had m.p. and mixed m.p. 156 to 157°C.

Anal. Calcd. for $C_7H_{14}O_6$: OCH_3, 16.0%. Found: OCH_3, 15.8%; $[\alpha]_D$ +50°C. (*c*, 2.5 in water).

Isolation of a Mixture of Hemicelluloses

Wood meal (1385 g.; 40–60 mesh) was suspended in water (14 liters) at 75°C. Glacial acetic acid (360 ml.) and sodium chlorite (120 g.) were added and the reaction was allowed to proceed with continuous stirring for 1 hr. (*19*). The same amounts of reagents were added every hour for an additional 3 hr.

The holocellulose was recovered by filtration, washed with water and ethanol, and dried in the air (962 g.; 69.6%). Holocellulose (720 g.) was shaken with 24% (w/w) aqueous potassium hydroxide (8 liters) at room temperature in an atmosphere of nitrogen for 10 hr. The alkaline solution was recovered by filtration through fritted glass and the solid residue was washed with water. The combined extract and washings (12 liters) were poured into a mixture of ethanol (30 liters) and acetic acid (3.5 liters) cooled to 0°C. The precipitate formed was recovered on the centrifuge, washed successively with 80% aqueous ethanol, ethanol, and petroleum ether, and dried *in vacuo* to give a white powder (155 g., 15.0%). The mixture contained five parts of xylan and three parts of glucomannan.

Preparation of Pure Hemicellulose

The mixture of hemicelluloses (154 g.) was dissolved in 10% aqueous sodium hydroxide (3 liters) and 5% aqueous barium hydroxide (5 liters) was added dropwise over a period of 3 hr. The precipitate formed was removed on the centrifuge and the supernatant solution was poured into a mixture of ethanol (25 liters) and glacial acetic acid (3 liters) cooled to 0°C. The precipitate was recovered in the usual way to give a white powder (135 g.). This treatment was repeated four times to yield a product (50 g.; 5% of the wood) which contained only xylose, arabinose, and uronic acid residues. The yields and composition of the five products are summarized in Table I.

A portion of the xylan (10.0 g.) was converted to the free acid form by treatment with ice-cold *N* hydrochloric acid (300 ml.) for 15 min. at room temperature. After centrifugation, the clear solution was poured into ethanol (1.2 liters) and the precipitate (8.0 g.) was recovered in the usual way.

Anal. Galactose, glucose, and mannose residues, nil; xylose/arabinose, 9.0; uronic anhydride, 15.1%; methoxyl, 2.8%; equiv. wt., 1000; $[\alpha]_D$ −65°C. (*c*, 1.0 in 5% sodium hydroxide).

Methylation of the Hemicellulose

The hemicellulose (10 g.) was dissolved in water (100 ml.) and 40% (w/w) aqueous sodium hydroxide (200 ml.) was added in an atmosphere of nitrogen, after which dimethyl sulfate was added dropwise over a period of 12 hr. This treatment was repeated four times. Solid sodium hydroxide (100 g.) and dimethyl sulfate (150 ml.) were added in the same way. This process was repeated four times, water being added whenever necessary for efficient stirring. The mixture was heated to boiling when the partly methylated product separated out and could be collected by filtration. The material was washed briefly with hot water and dried *in vacuo* to yield a hard powder (6.0 g.).

The polysaccharide was dissolved in dry dimethylformamide (300 ml.) and silver oxide (20 g.) and methyl iodide (20 ml.) were added to the clear solution. The mixture was shaken for 24 hr. This treatment was repeated twice. Chloroform was then added, insoluble salts were removed by filtration, and the solution (1000 ml.) was washed once with 5% aqueous potassium cyanide (200 ml.) and twice with water (200 ml.). After drying over sodium sulfate, the chloroform solution was concentrated to 100 ml. and added slowly to petroleum ether (800 ml.). The precipitate was washed with petroleum ether and dried *in vacuo* to yield a fluffy powder (5.1 g.)

Anal. Calcd. for the fully methylated and esterified hemicellulose: OCH_3, 39.6%; Found: OCH_3, 39.6%; $[\alpha]_D$ −60°C. (*c*, 1.0 in chloroform).

The infrared spectrum of the product indicated the absence of hydroxyl groups.

Methanolysis of the Methylated Hemicellulose and Separation of Acidic and Neutral Components

Methylated hemicellulose (4.0 g.) was boiled under reflux with 2% methanolic hydrogen chloride (100 ml.) for 12 hr. After neutralization and purification in the usual way, the sirup obtained (4.1 g.) was treated with 5% aqueous barium hydroxide (25 ml.) at 60°C. for 2 hr. (*22*). Solid carbon dioxide was added and barium carbonate was removed by filtration through Celite. After treatment with Amberlite IR-120 exchange resin, the acidic solution was added to the top of a column (3.5 × 18 cm.) of Dowex 1-X4 exchange resin (acetate form). Neutral sugars were eluted with water (4 liters), evaporation of which gave a clear sirup (2.40 g.). The acidic portion was displaced from the column with 30% aqueous acetic acid (4 liters) which was evaporated. The residual sirup was dried *in vacuo* at room temperature for 7 days. Paper chromatography (solvent A) indicated that the sirup (0.80 g.) contained only one component.

Preparation and Identification of Methyl 2-O-(2,3,-4-tri-O-methyl-α-D-glucopyranosyl)-3-O-methyl-α,β-D-xylopyranoside. The acid glycoside (800 mg.) was converted to its methyl ester which was reduced with lithium aluminum hydride (2.0 g.) in dry ethyl ether (50 ml.). The recovered sirup (710 mg.) crystallized spontaneously. After recrystallization from ethyl acetate, the crystals had m.p. and mixed m.p. 164 to 165°C. The infrared diagram of the compound was identical to that of an authentic sample of methyl 2-*O*-(2,3,4-tri-*O*-methyl-α-D-glucopyranosyl)-3-*O*-methyl-α,β-D-xylopyranoside (*16*).

Anal. Calcd. for $C_{16}H_{30}O_{10}$: OCH_3, 40.6%. Found: OCH_3, 40.5%; $[\alpha]_D$ +84°C. (*c*, 2.0 in water).

Separation of the Neutral Components of the Methylated Hemicellulose

The mixture of neutral glycosides (2.4 g.) was hydrolyzed with *N* sulfuric acid (50 ml.) by boiling under reflux for 6 hr. The solution was treated with Amberlite IR-120 and Dowex 1-X4 exchange resins and with a little Darco G-60 charcoal. Filtration and evaporation gave a mixture of reducing sugars (2.1 g.), a portion of which (1.0 g.) was added to the top of a charcoal (Darco-G-60)-Celite column (*23*) (3.5 × 55 cm.). Resolution was effected by gradient elution with the following solvents: water → 16% aqueous ethanol (2.5 liters each), 16% ethanol → 32% ethanol (2.5 liters), and 32% ethanol → 50% ethanol (1 liter). One 25-ml. fraction was collected every hour and every third of the 500 fractions obtained was examined by paper chromatography (solvent D). All five components in the mixture were completely separated. Experimental data are summarized in Table III.

Identification of 3-O-Methyl-D-xylose. This fraction

Table III. Resolution of the Neutral Part of the Methylated Sugar Mixture

Fraction no.	*Component*	*Weight, mg.*
91–96	3-*O*-Methyl-D-xylose	30
97–115	2-*O*-Methyl-D-xylose	110
148–189	2,3-Di-*O*-methyl-D-xylose	700
341–351	2,3,5-Tri-*O*-methyl-L-arabinose	75
356–360	2,3,4-Tri-*O*-methyl-D-xylose	15

had the same mobility on the paper electrophoretogram as an authentic sample of 3-*O*-methylxylose.

Anal. Calcd. for $C_6H_{12}O_5$: OCH_3, 18.9%. Found: OCH_3, 18.6%; $[\alpha]_D$ +20° (*c*, 1.3 in methanol).

The 3-*O*-methyl-*N*-phenyl-D-xylopyranosylamine had m.p. and mixed m.p. 135°C. (*11*).

Identification of 2-O-Methyl-D-xylose. The crystalline sugar, $[\alpha]_D$ + 35°C. (*c*, 1.0 in methanol), had m.p. and mixed m.p. 132 to 133°C. (*65*).

Anal. Calcd. for $C_6H_{12}O_5$: OCH_3, 18.9%. Found: OCH_3, 18.5%.

The 2-*O*-methyl-*N*-phenyl-D-xylopyranosylamine had m.p. and mixed m.p. 130 to 131°C. (*66*).

Identification of 2,3-Di-O-methyl-D-xylose. Paper electrophoresis indicated the absence of any 3,4-di-*O*-methylxylose in this fraction. The crystals obtained on seeding had m.p. 91 to 92°C. (*56*), $[\alpha]_D$ +26°C. (*c*, 1.0 in water).

Anal. Calcd. for $C_7H_{14}O_5$: OCH_3, 34.8%. Found: OCH_3, 34.4%.

The 2,3-di-*O*-methyl-*N*-phenyl-D-xylopyranosylamine had m.p. and mixed m.p. 124 to 125°C. (*67*), $[\alpha]_D$ +192 (*c*, 0.5 in ethyl acetate).

Identification of 2,3,5-Tri-O-methyl-L-arabinose. The sirupy sugar $[\alpha]_D$ −38°C., (*c*, 1.0 in water), had an R_f value slightly higher than that of 2,3,4-tri-*O*-methylxylose in solvent E.

Anal. Calcd. for $C_8H_{16}O_5$: OCH_3, 48.4%. Found: OCH_3, 47.5%.

The 2,3,5-tri-*O*-methyl-L-arabonamide, on recrystallization from methanol-petroleum ether, had m.p. 130 to 132°C. (*22, 68*).

Identification of 2,3,4-Tri-O-methyl-D-xylose. This fraction, $[\alpha]_D$ +20°C. (*c*, 1.5 in ethanol), crystallized on standing, m.p. and mixed m.p. 88 to 89°C. (*69*).

Anal. Calcd. for $C_8H_{16}O_5$: OCH_3, 48.4%. Found: OCH_3, 47.8%.

Selected Removal of L-Arabinofuranose Residues from the Hemicellulose

Hemicellulose (20 g.) was dissolved in 0.05 *N* aqueous sulfuric acid (100 ml.) and the pH was adjusted to 2.3, after which the solution was heated to 97° for 3 hr. The solution was poured into ethanol (4 liters) and the precipitate was recovered in the usual way to give a white powder (14 g.). Hydrolysis and examination by paper chromatography (solvents A and B) indicated the presence of xylose and uronic acid residues only.

Methylation of the Arabinose-Free Hemicellulose

The hemicellulose (10 g.) was dissolved in water (200 ml.) and sodium hydroxide (100 g.) was added in an atmosphere of nitrogen. Dimethyl sulfate (100 ml.) was added dropwise over a period of 18 hr. Three further methylations were carried out, each with addition of sodium hydroxide (200 g.), water (300 ml.), and dimethyl sulfate (200 ml.). Remaining alkali was neutralized with concentrated sulfuric acid and the reaction mixture was heated to boiling. The precipitate formed was collected and the remaining solution was exhaustively extracted with chloroform to yield an additional small quantity of material. The combined polysaccharides were dissolved in chloroform, reprecipitated into petroleum ether, and recovered to give a white powder (8.0 g.).

The partly methylated polysaccharide (7.0 g.) was dissolved in dry dimethylformamide (100 ml.) and silver oxide (50 g.) and methyl iodide (50 ml.) were added. After shaking at 25°C. for 2 days, the same amounts of silver oxide and methyl iodide were again added. After a further 3 days, the product was recovered in the usual way to give a white, fluffy powder (4.7 g.).

Anal. Calcd. for the fully methylated and esterified hemicellulose: OCH_3, 39.4%. Found: OCH_3, 38.4%.

The infrared spectrum indicated the absence of hydroxyl groups.

Methanolysis of the Methylated Polysaccharide and Removal of the Acid Fraction

The methylated hemicellulose (3.0 g.) was subjected to methanolysis as described above and the ester groups were removed by saponification. The acid glycoside was eliminated by adsorption on Dowex 1-X4 exchange resin (acetate cycle). The water eluate was evaporated to give a mixture of neutral glycosides, which was hydrolyzed to a colorless, clear sirup (1.8%).

Resolution of the Mixture of Methylated Sugars

The mixture of *O*-methylxyloses was added to the top of a Darco G-60 charcoal-Celite column (3.5 × 55 cm.) and resolved by gradient elution with 3 liters each of water → 15% aqueous ethanol and 15% ethanol → 30% ethanol. Three main fractions were obtained, namely a mixture of two mono-*O*-methylxyloses (50 mg.), a di-*O*-methylxylose (757 mg.) and a tri-*O*-methylxylose (16 mg.).

Characterization of the O-Methylxyloses. Paper electrophoresis suggested that the first fraction contained 2-*O*- and 3-*O*-methyl-D-xylose. The third fraction was chromatographically indistinguishable from 2,3,4-tri-*O*-methyl-D-xylose (solvents D and E). The second fraction, when recrystallized from ethyl acetate, had m.p. 94 to 95°C. undepressed on admixture with an authentic sample of 2,3-di-*O*-methyl-D-xylose, and $[\alpha]_D$ +22°C. (*c*, 2.0 in water).

Preparation of a Fully Acetylated Hemicellulose

Hemicellulose (1.0 g.) was shaken for 24 hr. with dry formamide (50 ml.). Freshly distilled pyridine (100 ml.) and redistilled acetic anhydride (20 ml.) were added and the reaction was allowed to proceed at room temperature with continuous shaking for 10 hr. (*35*). Acetic anhydride (40 ml.) was again added. After 15 hr., the mixture was poured with stirring into ice-water (1.5 liters) containing 2% hydrochloric acid. The precipitate was collected on the filter, washed with cold, distilled water until neutral, and then washed successively with ethanol and petroleum ether. The product was dried *in vacuo* to give a grayish, hard

Table IV. Osmometry Data

Methylated hemicelluloses				Acetylated hemicelluloses			
Original		Arabinose-free		Sample A		Sample B	
w[a]	h/w[b]	w	h/w	w	h/w	w	h/w
2.726	1.030	2.963	1.400	3.521	1.183	2.895	1.035
1.365	1.125	2.507	1.485	2.697	1.180	2.488	1.015
1.349	0.956	1.870	1.465	2.067	1.096	1.858	1.007
0.684	1.015	1.399	1.416	1.377	0.987	1.410	0.989
0.665	1.009	0.806	1.410	0.674	0.963	0.807	0.963
0	0.98	0	1.41	0	0.95	0	0.93

[a] Concentration in g./kg. solution.
[b] Osmotic height in cm. solution/concentration.

powder (1.2 g.). A duplicate acetylation of another hemicellulose sample gave a similar acetate in the same yield.

Determination of the Number-Average Molecular Weights of the Methylated and Acetylated Hemicellulose

Osmotic pressure measurements were carried out with the Zimm-Myerson (*70*) osmometers as modified by Stabin and Immergut (*71*). Gel cellophane membranes which had never been allowed to dry, the static method of measuring the osmotic height, and a temperature of 30°C. were used. The solvent was a 9 : 1 (v/v) mixture of chloroform and ethanol. The results obtained are summarized in Table IV.

Boundary Electrophoresis of the Hemicellulose (72)

A solution of the polysaccharide (1%) in 0.05*M* borate buffer was used, previously dialyzed against the solvent for 24 hr. The instrument used was a Spinco Model H apparatus. The polysaccharide moved as a single peak, thus indicating the presence of a homogeneous material.

Periodate Oxidation of the Hemicellulose

Portions of hemicellulose (150 mg.) were treated with 0.05*M* sodium metaperiodate in the dark for various lengths of time. The consumption of periodate was determined by the excess arsenite method. Oxidant consumed per repeating unit of the polysaccharide (*34*, *74*) was 20.5 moles.

Determination of the Weight-Average Molecular Weight of the Hemicellulose

Light-scattering measurements were carried out with a Brice-Phoenix photometer at a wavelength of 5460 A. The solvent was dimethyl sulfoxide containing 2% of water. The polymer solution (0.1%) was subjected to ultracentrifugation at 140,000 *g* followed by direct centrifugation in the light-scattering cell at 35,000 *g* (*36*, *73*, *74*). Scattering intensities were measured at 45, 90, and 135° relative to the incident beam, and at different concentrations. Corrections were not applied for dissymmetries, which were very small. The following values were obtained for $C/I_{90°}$ at the respective concentrations (mg./ml.): 3.040 (0.76), 4.702 (2,21), 6.594 (4.88), 8.184 (7.12), and 8.608 (9.12). Extrapolation to zero concentration gave a value of 3.30 for $(C/I_{90°})_{C=0}$. The refractive index increment was 0.0641 ml. per g. as determined at a wavelength of 4358 A. and at a temperature of 25°C. The molecular weight ($\bar{M}_w$) was calculated from the relationship $1/\bar{M}_w = H\,(C/\tau)_{C=0}$ where the symbols have their usual significance. The value obtained was 43,100 corresponding to a D.P. of 250.

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Variation in Some Wood Quality Attributes of One-Year-Old Black Cottonwood

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Samples of 1-yr.-old black cottonwood were divided into three groups with respect to their origin. Growth rate proved to have a statistically significant effect on fiber length and specific gravity. Faster growth was correlated with longer fibers and lower specific gravity. Clonal source influenced specific gravity independent of growth rate. Cellulose content was variable, but not associated with any of the factors tested.

This study was undertaken to determine the effects of growth rate, heredity, and site on fiber length, specific gravity and holo- and alpha-cellulose content in black cottonwood, *Populus trichocarpa* Torr. & Gray. The considerable differences among clones suggest the possibility of improving wood quality of black cottonwood substantially by breeding and selection within natural stands.

Evidence is increasing to support the suggestion that it is desirable to select fast-growing clones with high specific gravity, since specific gravity tends to decrease with increased rate of growth. Kennedy and Smith (*1*) showed that specific gravity in cottonwood increased significantly as rate of growth decreased. Swan (*2*) observed that density of poplar cuttings decreased by as much as 42% when growth rate was accelerated with gibberellic acid. Paul (*3*) reported that specific gravity of poplars decreased from 0.37 with a growth rate of 0.01 in. per yr. to 0.29 with annual growth rate of 1.0 in.

Kennedy (*4*), Kennedy and Smith (*1*), and the Carbondale Forest Research Center (*5*) have shown that fiber length increases significantly with growth rate. In a study of 83 cottonwoods, the Carbondale Forest Research Center concluded that measurable environmental influences accounted for 60% of the variability in fiber length, while the remaining 40% of the variation was attributed to individual tree differences probably genetically controlled.

No data are available to describe clonal variation in holo- and alpha-cellulose contents of black cottonwood.

MATERIALS AMD METHODS

All wood used in this study consisted of 1 yr.-old shoots grown from cuttings or seeds taken from individual black cottonwoods and cultivated in a nursery for West Tree Farms Ltd. All samples were about 15 in. long and were cut from 5 to 15 in. above the ground, depending on the form and growth rate of the shoot.

From each sample a section 2 to 5 in. in length was cut, peeled, measured as to diameter, split, and quartered after removal of the pith. One quarter was used for determination of fiber length, another for specific gravity, a third for cellulose, while the fourth served as a spare.

Pulp slides were prepared according to previously reported techniques (Kennedy, *4*). In taking cell length measurements, 40 randomly chosen fibers from each sample were measured to the nearest 0.01 mm. from images projected onto a graduated target using the method described by Wilson (*6*).

Specific gravity (green volume) was determined for each specimen by the maximum-moisture technique of Keylwerth as outlined by Smith (*7*).

Holocellulose and alpha-cellulose were determined by a modification of the chlorite method originally suggested by Wise *et al.* (*8*). This modification was developed by Yundt and Bradway for the use of Zobel and McElwee in evaluating loblolly pine (9). It has also been used by Kennedy and Jaworsky (*10*) for Douglas-fir.

RESULTS AND DISCUSSION

The basic data are given in Table I for three groups. The first contains three clones of black cottonwood

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grown on clay. The second compares cuttings, seedlings, and suckers grown or cut from a single tree. The third comprises seedlings of a single tree grown on sand and on clay. Each source of material is represented by two fast-grown and two slow-grown 1-yr.-old shoots. Diameter inside bark is also given as a measure of growth rate. For each replicate, fiber length in millimeters and green volume specific gravity are tabulated. In groups I and II, averages of three replicates of holocellulose and two replicates of alpha-cellulose estimates are given for each growth rate class (two samples). Each sample was measured similarly for per cent cellulose in group III.

cuttings and seedlings and between clay and sand were not significant as reflected by specific gravity.

The high specific gravity and alpha cellulose content of the suckers is probably due to the presence of tension wood. Microscopic examination of cross sections from the suckers revealed masses of gelatinous fibers. Lassen (*11*) has indicated an increase in specific gravity and Schwerin (*12*) an increase in alpha-cellulose contents of tension wood. Tension wood is more likely to be present in suckers than in stems propagated from seeds or cuttings because of their leaning habit in growing away from the bole of the parent.

Apart from the suckers, yields of holocellulose and alpha-cellulose were not associated significantly with any of the factors tested.

Table I. Summary of Data Collected on 1-Yr.-Old Black Cottonwood

Group	Method of propagation	Source of material	Rate of growth	Diam. inside bark, mm.	Mean fiber length, mm.	Specific gravity (green volume)	Cellulose, % Holo-	Cellulose, % Alpha-
I	Cuttings	Tree 57-13	Fast 1	13.0	0.66	0.367	81.5	42.4
			Fast 2	7.5	0.59	0.388		
			Slow 1	6.0	0.56	0.397	85.7	43.4
			Slow 2	5.5	0.56	0.439		
	Cuttings	Tree 57-12	Fast 1	21.5	0.69	0.352	82.4	44.9
			Fast 2	18.5	0.72	0.354		
			Slow 1	11.5	0.57	0.361	81.4	44.2
			Slow 2	9.0	0.57	0.397		
	Cuttings	Tree 57-3	Fast 1	23.5	0.81	0.400	83.2	43.8
			Fast 2	22.5	0.77	0.404		
			Slow 1	9.0	0.59	0.426	82.7	43.5
			Slow 2	7.0	0.55	0.462		
II[a]	Seedlings	Tree 57-13	Fast 1	8.5	0.54	0.363	82.3	43.2
			Fast 2	8.0	0.56	0.388		
			Slow 1	7.0	0.57	0.383	79.4	39.8
			Slow 2	4.5	0.45	0.407		
	Suckers	Tree 57-13	Fast 1	7.8	0.57	0.423	84.5	50.1
			Fast 2	6.7	0.51	0.445		
			Slow 1	4.6	0.57	0.473	81.7	48.2
			Slow 2	3.4	0.60	0.477		
III	Seedlings grown on sand	Tree 57-16	Fast 1	12.0	0.63	0.380	81.2	40.3
			Fast 2	10.0	0.59	0.413	81.9	42.8
			Slow 1	4.5	0.53	0.417	79.3	39.8
			Slow 2	4.0	0.52	0.417	81.6	40.7
	Seedlings grown on clay	Tree 57-16	Fast 1	18.0	0.62	0.394	83.8	42.8
			Fast 2	16.5	0.62	0.414	81.5	42.7
			Slow 1	8.5	0.58	0.408	83.7	45.0
			Slow 2	7.0	0.56	0.434	81.2	42.9

[a] Group II includes 57-13 in group I.

With the exception of the suckers from 57–13, fiber length increased significantly with growth rate. In the group I data the correlation coefficient for the increase of fiber length with diameter growth rate is highly significant, $r = 0.95$. No important clonal influence on fiber length other than that caused by the fast growth of 57–3 is present. At comparable rates of growth, no significant differences in fiber length existed between cuttings and seedlings from tree 57–13. The atypical nature of the sucker material is discussed below.

Specific gravity within each clonal and seedling group decreased significantly as growth rate increased. The possibility of selection to improve specific gravity is strikingly clear in comparisons of clones 57–12 and 57–3. These had similar average growth rates of 15.1 and 15.5 mm. but significantly different specific gravities of 0.366 and 0.423, respectively. In spite of the fact that, within a single clone, specific gravity increased as growth rate decreased, clone 57–13 with a slow growth rate of 8.0 mm. had a specific gravity of only 0.398 on the average. This indicates that growth rate may not be reliable in predicting specific gravity of genetically variable material. Differences between

CONCLUSIONS

Where age differences are eliminated and environmental conditions are described as in this study, it is possible to show some highly significant relationships controlling quality of black cottonwood. Fiber length increases and specific gravity decreases as growth rate increases. Only specific gravity appears to be strongly controlled genetically. This is fortunate since the advantages of selection or breeding for rapid growth of high density wood for pulping are obvious.

Clone 57–3 should be propagated for its apparent ability to produce wood of high specific gravity and long fibers when fast grown.

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Development of a New Retention Aid

LLOYD O. BOOTS

This is an investigation of Nalco 600-D1593/mixture as a paper machine retention aid. The Nalco mixture as a retention aid is based on the electrokinetic theory of particles carrying opposite charges attract each other. The project compared this new retention aid with some other retention aids for efficiency as measured by headbox and tray water consistencies and on a total cost per ton of paper basis. A part of the study consisted of evaluating the increased efficiency of the titanium dioxide retained in the sheet with regards to brightness, opacity, and "two sidedness." A limited amount of work was done on colored paper with the emphasis being on minimizing wide color formula variations from day to day, increased color retention, and color uniformity from felt to wire side of sheet.

According to Webster "Retention"—The act of holding or continuing to hold in possession or use. Retention of something of value—whether it be money, jewelry, or that box of junk in the basement—appeals to all of us. It is also true with papermaking raw materials.

The aim in papermaking is to make a salable, quality product with the minimum amount of raw materials. If this aim is to become a reality it is probable that some form of retention aid will be utilized. Retention of cellulose fines and fillers (clay, titanium dioxide, talc, and other materials) to a large extent, has been accomplished by means of filtration. The appropriate parts of the fourdrinier section act as a vacuum pump while the forming sheet of paper is the filter media. Many retention aids act as coagulants by increasing the size of fines and filler particles. It would seem that the ultimate in filler utilization and retention would be reached when the individual filler particles are bonded to the individual fibers.

The electrokinetic potentials of cellulose fibers, filler particles, size precipitate, and other particles in the headbox slurry of a paper machine have a great effect on the formation of the sheet on the fourdrinier wire.

If the electrokinetic potentials of the materials have the same sign they repel each other. Thode and Htoo in *Tappi* 38, No. 12 (December, 1955) determined that when cellulose fibers are dispersed in water they have a highly negative potential, the sign of which is extremely difficult to reverse. Most fillers are also anionic although their potential is not considered to be as strong. If the charge on the filler could be changed and/or fortified then the bonding of filler to fiber would seem a probability. This was the motivating theory behind the development of the Nalco 600-D1593 retention aid.

Nalco 600 is a polyamine-type cationic material which was developed as a coagulating aid for water treatment plants. Since it is cationic and it has an attraction for cellulose fibers, it was tried by itself as a retention aid on paper machines but it was not outstanding. Laboratory trials were then run to try and evaluate the possibility of adding "600" to the fiber and an anionic material to the filler. These trials showed some promise but it was not always practical to treat the materials in this way and an evaluation of a mill trial of short duration would be extremely difficult. Mixing the Nalco 600 with an anionic was tried for compatability and as a possible retention aid. This approach seemed rather promising and various anionic materials at differing ratios of mixture were tried. It finally evolved that Nalco D1593 was the most effective anionic and that a mixture of 1 part of the anionic to 200 parts of Nalco 600 by weight was the proper ratio.

When the composition of the retention aid had been established, the various addition rates were investigated. A laboratory beater was used for refining and mixing. A charge of 450 g. of deinked stock was added to the beater and it was refined to a Schopper-Riegler freeness of 550. After refining, the weights were removed from the beater and the balance of the furnish was added as follows: 45 g. or 10% TiO_2 67.5 g. or 15% filler clay, 3.5 g. or 0.8% rosin size, and finally alum to pH of 5.0. Handsheets were made on a Valley sheet mole using 3.2 g. of stock. The various amounts of retention aid were added to the sheet mold and the suspension gently agitated for 15 sec. to insure mixing. The sheets were dried on stainless steel plates after which the brightness, basis weight, and per cent ash was checked. The following table summarizes the results.

Retention aid and amount		*Sheet weight, lb.*	*G.E. brightness*	*Ash, %*	*Retention, %*
Control none	lb.	54.3	80.0	6.7	27.0
Nalco 600-D1593	2 lb./ton	54.7	81.0	8.6	34.4
Nalco 600-D1593	3 lb./ton	55.2	81.0	9.8	39.2
Nalco 600-D1593	4 lb./ton	56.0	82.0	10.5	40.2
Sveen glue	2 lb./ton	55.0	81.0	8.8	35.2

Laboratory results indicated a 15% increase in basis weight should be expected when adding 4 lb. per ton of Nalco 600-D1593 retention aid if no retention aid was

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used previously. A 5% increase in basis weight should result if it replaced 2 lb. per ton of glue. Machine trials have followed these laboratory results rather closely.

DISCUSSION

The following descriptions and tables summarize the machine trials of this new retention aid. A trial run of Snowfield high brightness offset was made on the No. 2 machine. Since this grade is not produced normally on this machine, a minimum quantity of 50, 80, and 100 lb. was made as a means of evaluating certain mechanical problems that were believed to exist. A trial of the special retention aid was started with the Snowfield run. This production was preceded by a run of low brightness paper (72 to 73 G.E. brightness) and the change to the high brightness sheet was made without flushing out the system. A total of 400 lb. of titanium dioxide per ton was added to the furnish and a brightness of 88 to 88.5 G.E. was desired. In the transition from 72 brightness, the hoped for brightness of 88 was merely a passing phase on the way to an ultimate brightness of 90.5 felt and 90.1 wire side. Two 2000-lb. reels of each weight paper were made with the shade or color matching a 70 sheet previously made on the no. 1 machine. Neither the shade nor the brightness varied from the beginning to the end of the run in spite of the weight and machine speed changes. Normally an appreciable difference in ultraviolet reflectance is shown between the two sides of a high brightness sheet. This is caused by the ultraviolet absorption properties of titanium dioxide for fluorescent dyes. Since titanium dioxide is normally concentrated on the top side of the sheet, it would show less ultraviolet reflectance from the optical brighteners used in the surface size than the reflectance shown from the wire side. The ultraviolet reflectance reaffirmed the evenness of the titanium dioxide retention by showing little difference to the "black light" on either side of the sheet. While the comparative 70-lb. sheet was made on a different paper machine than the 50 and 100-lb. sheets, the "wet ends" and speeds of the two machines are very similar. The comparative tests, and the titanium dioxide and fluorescent dye furnish are as shown.

	Regular—glue	*Nalco 600-D1593*	*Nalco 600-D1593*
Weight, lb.	70	50	100
Brightness	88.5	90.5	90.5
Ash, %	10.0	15.0	15.5
Opacity	96	96	99
TiO_2, lb./ton	400	400	400
Florescent dye, lb./ton	7	7	7

The latest machine trials of this retention aid are shown by the following table. The material was used on two consecutive days on the no. 3 paper machine. The per cent retention for both trials was identical. The machine speed on these trials was 1020 f.p.m. producing a 50-lb. sheet. The opacity of the sheet with the 600-D1593 increased slightly over the sheet using glue as a retention aid although the per cent retention of titanium dioxide dropped from 96 to 89%. The titanium dioxide charge was three per cent based on the pulp so that any slight change in titanium dioxide retention would be apparent. The increase in opacity with a decrease in retention efficiency points out the fact that retention alone is not enough but the method of retention is extremely important.

Retention aid		*Consistency—Headbox*	*Consistency—Tray*	*Ash, %—Total available*	*Ash, %—Reel*	*Retention, %*
Glue	2 lb./ton	0.695	0.384	13.8	8.7	63.0
600-D1593	4 lb./ton	0.680	0.347	16.9	11.3	69.5
Glue	2 lb./ton	0.763	0.441	18.2	11.9	65.5
600-D1593	4 lb./ton	0.729	0.334	18.2	12.6	69.5

Any new product is surrounded with problems—the Nalco 600-D1593 is no exception. This retention aid has been under investigation at Simpson Paper Co. for the past 14 months. Numerous problems have been encountered and at the present time many of them remain unsolved. Most of the problems have concerned the anionic material. The D1593 is a high molecular weight acrylic material in a very fine powder. It is soluble in warm water (185°F.) but it does not wet out very readily. The first mill trials of relatively short duration were encouraging. When the retention material was tried over a longer period of time, its efficiency diminished after 4 or 5 hr. As the Nalco 600 and the anionic is mixed, a bonding or chemical reaction occurs. If the anionic is not completely in solution, the reaction seems to be reversible. When this happens, the mixture loses at least one-half of its effectiveness. When the retention aid is formulated, the anionic material in solution must be added to the Nalco 600 and with proper agitation. If the reverse addition order is tried, the D1593 forms a sticky mucus-like jell. Another problem that was encountered was a very distinct increase in shade difference between the felt and wire side of the sheet during both canary and buff color runs. This problem was extremely frustrating since it followed an excellent run of both blue and pink paper. After lengthy discussions with dye company technicians, it is suspected that the furnish was not properly dyed before it reached the paper machine. Further laboratory tests indicate that the Nalco 600-D1593 has no adverse effects on color shade after the color has been exhausted on the fiber. This conclusion must be verified on a future machine trial.

CONCLUSIONS

Nalco 600-D1593 looks very promising as a retention aid that bonds through electrokinetic attraction. It shows an improved drainage rate on the machine, better distribution of filler, more even and better retention of color, good stable bonding of filler to fiber, but it still offers some unknown quantities. Some of the uncertain aspects are: best point of addition; true shelf-life of the combined material—whether it is days, weeks, or months; ideal pH; effect of various amounts of rosin size; ideal aluminum ion concentration; best headbox temperatures; how much is carried over to the saveall; better ways to make the final product. These and many other questions must be resolved in the future. Because of these unmet challenges, this paper can be only a progress report—the final chapters are still to be written.

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Studies on the Mechanism of Wet Strength Development. II

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Studies are presented on the general charactetistics of urea-formaldehyde resin treated papers and on the rate of loss of wet strength of these papers when immersed in distilled water at various temperatures. The initial rates lead to an activation energy of 23 kcal. per mole for both cotton and unbleached kraft papers. It is shown that the cross-linked resin becomes progressively more soluble in the water on soaking. The results are consistent with the loss of wet strength being due to hydrolytic breakdown of the resin causing water solubility. The hydrolysis reaction appears to be the reverse of the curing reaction, and for each temperature and relative humidity, there exists a well-defined equilibrium between these reactions. Model compound studies indicate that cellulose hydroxyls do not form chemical linkages with the polymerized UF-resin. Some comparative observations are reported on neoprene and urea-formaldehyde wet strength papers.

Previous studies (*1*, *2*) have indicated that the formation of wet strength in papers treated with phenolic as well as urea-formaldehyde resins is due to the second stage condensation of the resin to a cross-linked insoluble polymer. The mechanism of wet strength formation is not known in detail; however, the results obtained in this laboratory (*2*) are compatible with the hypothesis that the wet strength is largely due to the protective action of the resin on the interfibrillar hydrogen bonds. Similar interpretations have been given by other workers in the field (*3*, *4*). A necessary condition for high degrees of wet strength may be the penetration of the resin into the amorphous cellulose of the interfibrillar regions.

The present paper describes some additional experiments concerning the properties of urea-formaldehyde treated papers and the loss of wet strength of these papers by prolonged immersion in water.

EXPERIMENTAL

The cotton and kraft papers had the characteristics described in a previous publication (*2*), where the saturation and curing techniques of the wet strength papers have also been described. Neoprene papers were prepared by carrying out the impregnation in various dilutions of Neoprene latex type 735 (37% solids, E. I. du Pont de Nemours and Co.). The Instron tensile tester was used for the tensile strength measurements, both under dry and wet conditions, as well as for the determination of stress-strain curves. The distance between the cross-heads was 4 cm. and the straining speed was 10 in. per min.

Experiments to determine the degradation of wet strength by immersion in water were carried out in a constant temperature bath filled with distilled water and equipped with a heating coil and a stirrer. Strips of paper, cut to the proper size for testing, were placed between two stainless steel screens and submerged in water by hanging the screens on a supported bar. For testing, 5 to 10 strips were taken out, the surface water removed by putting them between two blotters for a few seconds, and the wet strength was determined as an average of the individual tensile strength measurements. Generally, the wet tensile strengths were found to be less scattered than the dry-strength values.

The water-extraction experiments were carried out in the following way: The resin-treated and cured paper was soaked in distilled water and disintegrated in a Waring Blendor. The resulting slurry (2% consistency) was kept at 45° for 24 hr., filtered and dried. The resin content in the original as well as in the extracted sample was determined by Kjeldahl nitrogen determination. The same procedure was repeated with the extracted sample for subsequent extractions.

COMPARISON OF WET-STRENGTH PROPERTIES OF PAPERS SATURATED WITH UREA-FORMALDEHYDE RESIN AND NEOPRENE LATEX

Neoprene latices have been gaining in interest in recent times for the preparation of wet-strength papers. Both the chemical and physical properties of neoprene differ significantly from those of urea-formaldehyde resins. Whereas the latter are essentially hydrophilic polymers containing functional groups which react to form rigid cross-linked structures, neoprene is a hydrophobic, nonreactive, and highly extensible polymer. In view of such differences, it seemed of interest to study the manner in which the polymer properties are reflected in the properties imparted to saturated papers by these polymeric materials.

Comparative experiments, illustrated by Table I and Figs. 1 and 2, demonstrate that significant differences do indeed exist between urea-formaldehyde- and neoprene-treated papers. Whereas the former papers exhibit practically constant wet- to dry-strength ratios regardless of the kind of paper used, the wet strength imparted

Table I. Comparative Wet-Strength Values of Urea-Formaldehyde (UF)- and Neoprene-Treated Papers

	Tensile strength, Kg. per 15 mm. width					
	Neoprene impregnated[a]			*UF-Resin impregnated*[b]		
Paper	*Dry*	*Wet*	*Wet strength, %*	*Dry*	*Wet*	*Wet strength, %*
Cotton	5.2	1.64	32	4.2	2.18	52
Filter	3.9	1.59	41	5.9	3.1	53
Unbleached kraft	7.4	0.82	11	9.2	4.9	53

[a] Neoprene content: 2.5%, cured at 105°C. for 5 min.
[b] Impregnated in a bath containing 5% of unmodified UF-resin and cured at 120°C. for 12 hr.

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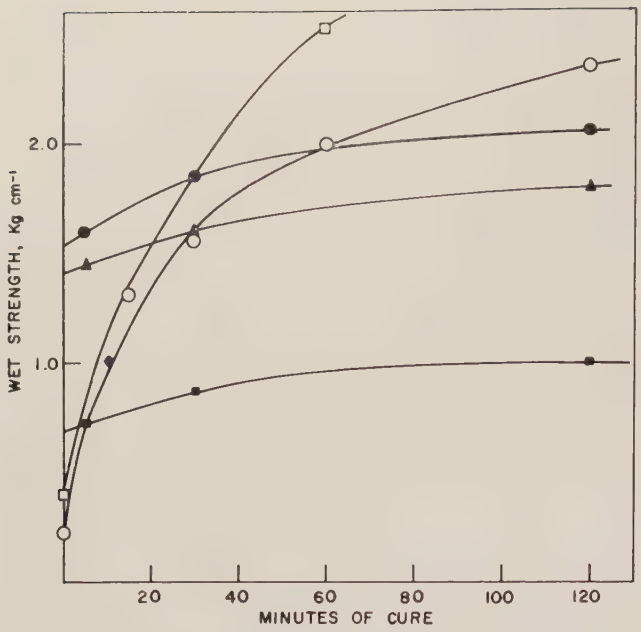


Fig. 1. Wet strength versus time in neoprene- and urea-formaldehyde-saturated papers

● Cotton paper. ▲ Filter paper. ■ Kraft paper. ⊙ Cotton paper UF-Resin content = 9.9%. Curing temperature = 140°C. ⊡ Kraft paper UF-Resin content = 12.1%. Curing temperature = 120°C. Neoprene content = 2.1%. Curing temperature = 105°C.

by neoprene is highly dependent on the base-paper used (Table I). Especially noteworthy was the low wet strength of neoprene-treated kraft papers, whereas cotton papers appear to respond especially favorably to neoprene saturation.

The urea-formaldehyde results are in line with the widely accepted assumption that wet strength results from the protective action of the resin on the pre-existing bonds of the fiber. The reason that the kraft paper in the case of neoprene gives the lowest strength is probably due to the inherent hygroscopicity of the

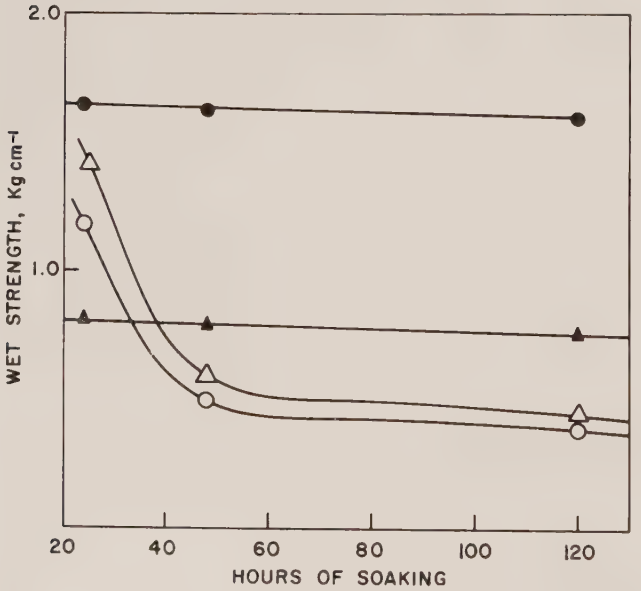


Fig. 2. Wet strength as a function of soaking time in water at 25°C.

● Cotton paper. ▲ Kraft paper. ○ Cotton paper. △ Kraft paper. Neoprene content = 2.5%. UF-Resin content = 4.1%.

fiber surfaces compared with cotton fibers. This favors the hydrophilic, low molecular weight, reactive urea-formaldehyde resin, but leads to a rapid penetration of water in the case of neoprene causing a considerable loss of strength on soaking in water.

The difference between the urea-formaldehyde- and neoprene-treated papers is also reflected in their elonga-

Table II. Per Cent Elongation at Break

	Base paper		Neoprene impregnated[a]		UF-Resin impregnated[b]	
	Dry	Wet	Dry	Wet	Dry	Wet
Cotton	2.10	0.57	4.00	4.50	2.80	4.50
Filter	1.89	1.28	4.10	8.90	2.20	4.05
Unbleached kraft	2.50	0.71	3.50	3.40	2.20	4.40

[a] Neoprene content = 2.1%.
[b] UF-Resin content = 5.1%.

tion values. The wet elongation at break of cotton and filter papers is roughly twice the elongation of the corresponding urea-formaldehyde treated papers. The wet elongation of neoprene-treated kraft, however, is conspicuously low, lower even than the value of urea-formaldehyde-treated kraft paper. This sort of behavior would perhaps be expected, if the protection of the fiber-fiber bonds in kraft was inferior to cotton as discussed earlier.

The effect of curing on neoprene- and urea-formaldehyde-treated papers (Fig. 1) is also essentially what could be expected on the basis of the properties of these polymers. Because of the hydrophobic nature and high molecular weight of neoprene, papers saturated with this polymer possess high wet strength already in the uncured state. The increase in wet strength is relatively small compared with urea-formaldehyde papers in which the polymerization process is the essential factor for producing wet strength.

Neoprene papers retain effectively their wet strength under prolonged soaking in water, whereas the stability of urea-formaldehyde under such conditions is substantially less (Fig. 2). The reason for the latter phenomenon is not immediately obvious and prompted a closer study to elucidate the factors involved.

Effect of Prolonged Soaking and Relative Humidity on the Wet strength of Ureaformaldehyde-Treated Paper

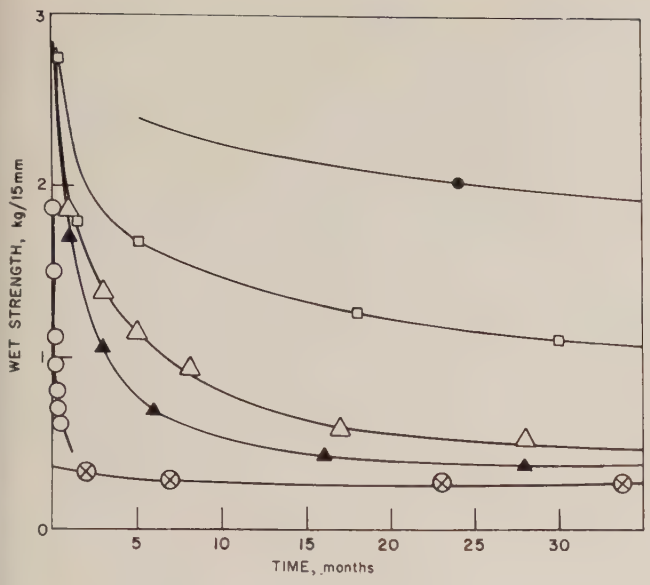
The wet strength degradation of paper treated with urea-formaldehyde resin is highly dependent on temperature (Figs. 3 and 4). As the temperature is increased, the rate of the process is greatly accelerated,

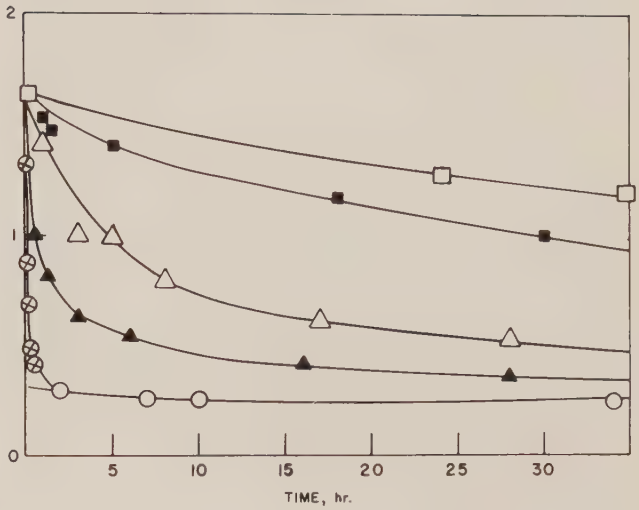
Table III. Removal of Resin from UF-Resin Treated Papers by Water Extraction at 45°C. Time of Each Extraction—24 Hr.

	Type of paper					
		Filter paper				Kraft
Curing time, hr. →	0	2	6	24	4	24
Curing temperature, °C→		105	105	105	140	105
No. of extractions	Resin in paper after extraction, %					
0	4.36	4.36	4.40	4.20	3.72	3.91
1	1.48	2.28	2.57	3.19	3.46	0.69
2	1.00	1.59	1.76	2.02	2.58	0.62
5	0.55	0.92	1.10	1.24	1.00	0.54
10	0.48	0.49	0.69	0.80	0.66	0.58

and the wet strength rapidly decreases and eventually reaches almost the level of the untreated paper. In boiling water, the paper totally loses its wet strength in about one hour.

The approximate activation energy of the overall





Figs. 3 and 4. The loss of wet strength with time on soaking in distilled water at various temperatures

● 23°C., □ 35°C., △ 55°C., ▲ 65°C., ○ 100°C. ⊗ Untreated paper kept at 120°C. for 2 hr. immersed in water at 23°C. Resin content = 5.3%; cured at 120°C. for 2 hr.

Resin content = 4.2%; □ 23°C., ■ 35°C., △ 55°C., ▲ 65°C., ⊗ 100°C. ○ Untreated paper kept at 120°C. for 2 hr. immersed in water at 23°C.

degradation process was estimated from the slopes of the curves in Figs. 3 and 4, in a manner similar to that used in previous papers for the rate of formation of wet strength on curing (*1*, *2*). Since the initial wet strength values are somewhat uncertain, owing to the time-lag, until complete wetting is achieved, the slopes at an arbitrary wet strength value of 1.30 kg. per 15 mm. were chosen as basis for constructing the Arrhenius plot in Fig. 5. It can be seen that the data on kraft and cotton papers both fall essentially on the same straight line. The activation energy, calculated from the slope of this line, is approximately 23 kcal. per mole.

One could tentatively consider two alternative possibilities to explain the decrease in wet strength by the action of water. Firstly, the resin may give no permanent protection to interfibrillar hydrogen bonds, but only retard the penetration of water into the bonding regions. The wet-strength decrease would then be due to a slow diffusion of water, followed by the solvolytic breakage of hydrogen bonds. The activation energy for such a process, however, would be of the same order of magnitude as found for the diffusion of water through plastic membranes, namely 10 kcal., or less. The experimentally found activation energy strongly discounts this possibility.

Secondly, the process could be governed by a hydrolytic cleavage of the cross-linked resin. Such a hypothesis is compatible with the activation energy found, and is further supported by the fact that increasing amounts of the resin become soluble in the aqueous phase dur-

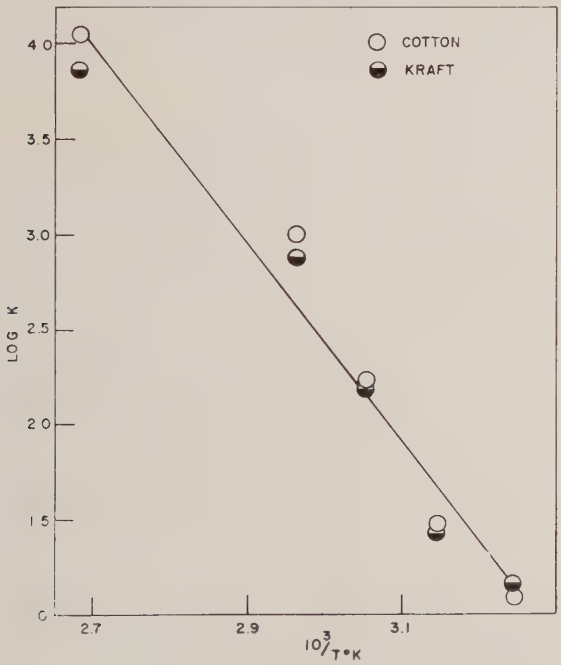


Fig. 5. Temperature dependence of the initial rates of degradation of wet strength by the action of water for urea-formaldehyde resin treated cotton and kraft papers

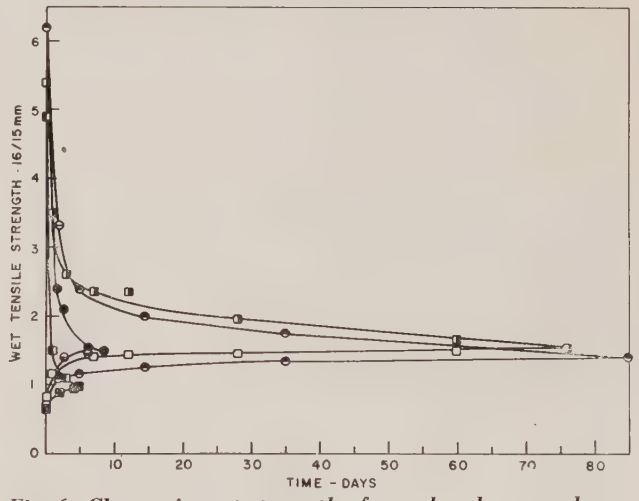


Fig. 6. Change in wet strength of cured and uncured urea-formaldehyde papers kept at 100% R.H. for various periods of time at 45 and 65°C.

Cotton paper: ◓, 6.0% UF-resin, uncured, heating at 45°C. 100% R.H. ◓ 6.0% UF-resin, cured at 120°C. for 6 hr. heating at 45°C. 100% R.H. ● 6.0% UF-resin, cured at 120°C. for 6 hr., heating at 65°C. 100% R.H. ◒ 6.0% UF-resin, uncured, heating at 65°C. 100% R.H.

Kraft paper: ◘, 4.3% UF-resin, cured at 120°C. for 6 hr., heating at 45°C. 100% R.H. □ 4.3% UF-resin, uncured, heating at 45°C. 100% R.H. ◧ 3.8% UF-resin, cured at 120°C. for 6 hr., heating at 68°C. 100% R.H. ■ 3.8% UF-resin, uncured, heating at 68°C. 100% R.H.

ing the water treatment and most of it can ultimately be removed from the fibers by successive washings (Table III). It seems probable, therefore, that the decrease in wet strength is due to a chemical reaction which is essentially the reverse of the curing reaction, i.e.:

$$\text{Urea-formaldehyde resin} \underset{\text{wet-strength degradation}}{\overset{\text{curing}}{\rightleftharpoons}} \text{Cured urea-formaldehyde resin} + H_2O$$

The reality of the postulated equilibrium and its influence on the wet-strength properties of the paper was demonstrated in the following manner. A batch of urea-formaldehyde treated papers was divided in two parts, one part was thoroughly cured at a high temperature, the other part was left uncured. Samples

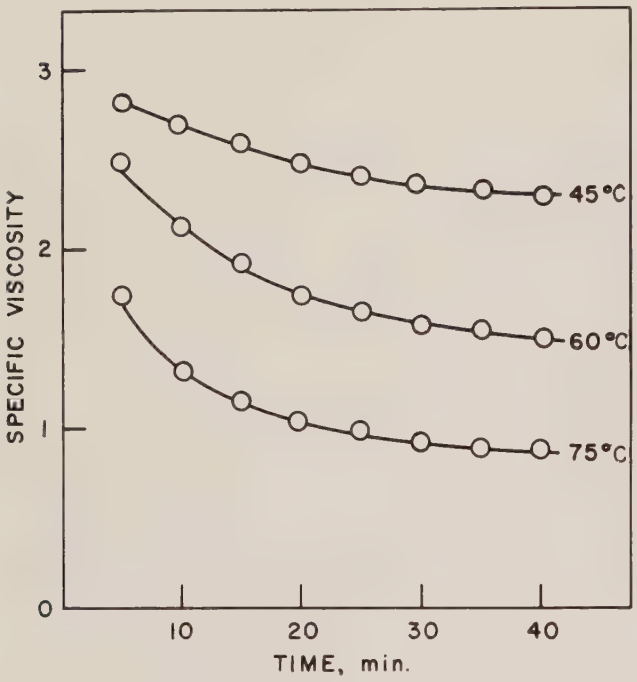


Fig. 7. Decrease in viscosity of a UF-resin solution at various temperatures. 20% unmodified UF-resin solution at pH 6.3

of both cured and uncured papers were kept for various periods of time at 100% R.H. in a closed desiccator immersed in a thermostat. The measured wet-strength values as a function of time are given in Fig. 6.

It can be seen that regardless of whether the papers were initially cured, their wet strengths approach gradually an identical value. This suggests that, for each level of relative humidity and temperature, there exists a well-defined polymerization-depolymerization equilibrium of the urea-formaldehyde resin. This equilibrium apparently determines the final wet strength attainable in each system. Uncured papers approach this equilibrium through a process of slow polymerization, whereas cured papers become the subject of gradual hydrolytic depolymerization. It was possible to observe the occurrence of the latter reaction in aqueous urea-formaldehyde resin solutions (Fig. 7). When heated at constant temperatures, the viscosity of these solutions decreased constantly, indicating a hydrolytic degradation of the dissolved resin.

The rate of approaching the equilibrium condition is probably not only dependent on temperature, but can be influenced by the pH of the solid phase which, in turn, is dependent on acidic or basic components in paper (e.g., partially neutralized lignosulfonic acid in strong sulfite or residual alkali in kraft papers). These components, of course, can also influence the rate of loss of wet strength by soaking in water. However, no detailed studies were carried out on this point.

Uncured urea-formaldehyde-treated paper gains slowly in wet strength during prolonged storage at room temperature (Fig. 8). It seems reasonable to ascribe the increase in wet strength to slow polymerization of the uncured resin in the paper.

The practical implication of the above studies is that the wet strength of urea-formaldehyde-treated papers is not an invariable characteristic dependent on the

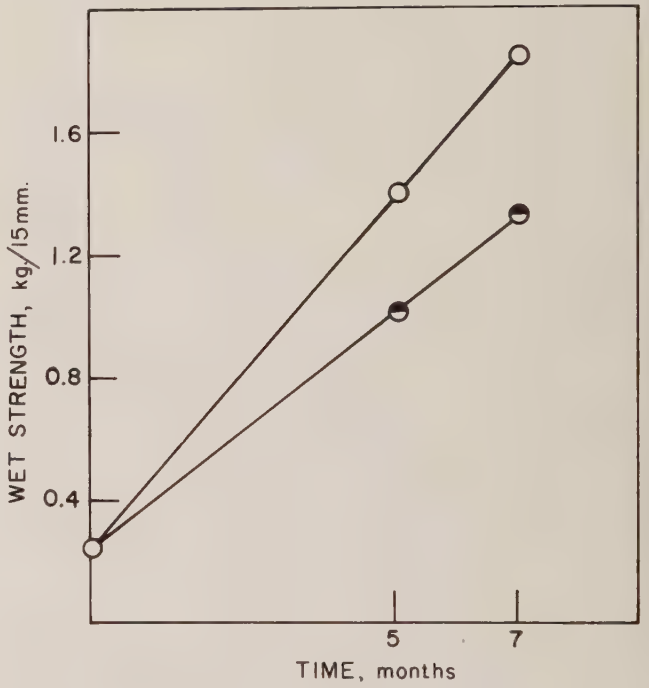


Fig. 8. The development of wet strength in uncured urea-formaldehyde resin treated kraft paper during storage at 23°C. (73°F.)

◓ Resin content = 5.3%. ○ Resin content = 12%.

resin content and completeness of curing, but is subject to changes caused by the storage conditions. Prolonged storage under humid conditions may slowly decrease the wet strength, whereas paper kept in dry atmosphere may show the opposite trend. The second implication is the importance of carefully specifying, in wet strength determinations, the duration and temperature of the soaking operation.

THE EXISTENCE OF CHEMICAL BOND BETWEEN UREA-FORMALDEHYDE RESIN AND CELLULOSE

Much speculation has been devoted, in the past, to the question whether chemical links exist between the cured resin and the carbohydrate components of wet-strength papers. Since the amorphous carbohydrates and the resin form an interpenetrating system, it would seem plausible that the hydroxyl groups of the former, if reactive, may enter in an ether linkage with the resin.

How the resin-carbohydrate bonds would affect the rheological properties of saturated papers cannot be visualized at the present stage of knowledge. On the other hand, the reactivity of carbohydrate hydroxyls with urea-formaldehyde (UF) resin lends itself to straightforward testing by the aid of model compounds. As such, methyl-α-glucopyranoside was selected as a model for carbohydrates and its reactivity tested toward unmodified UF resin solutions as well as towards a model compound of the latter, 1,1-dimethyl-3-methylol urea.

Table IV. Reactivity of Alpha-Methyl-Glucoside Toward Urea-Formaldehyde (UF) Resin and Its Model Compound (1,1-Dimethyl-3-Methylol Urea)

General conditions: pH adjusted to 4.0; heating temperature 105°C.

Test no.	*Amount of UF-resin soln.[a] added to 5 cc. of glucoside (100 g./l in H_2O)*	*Heating conditions*	*Amt. of insoluble cross-linked product formed*	*Glucoside recovery in soln. (based on rotation), %*
1	0 (blank)	12 hr. In sealed tube	Nil	100
2	1.5 cc.	12 hr. In sealed tube	Nil	99
3	2.5 cc.	12 hr. In sealed tube	Nil	99
4	1 g. of model compd.	15 hr. In sealed tube	Nil	98
5	2.5 cc.	12 hr. In sealed tube	Complete conversion	99
6	5.0 cc.	12 hr. In open tubes	Complete conversion	98

[a] Unmodified urea-formaldehyde solution (40% solids).

The first set of experiments (Table III) was based on the principle that a reaction between the glucoside and UF resin might be manifested in the changed optical rotation of the former compound. Experiments carried out by heating in sealed tubes the methyl glucoside in a concentrated aqueous solution containing UF resin or the model compound did not result in detectable changes in the optical rotation. These experiments did not lend support to the idea of mutual reaction, but neither did they clearly disprove the possibility of such reactions.

To obtain an unambiguous solution to the problem, the approach was slightly modified. Heating the glucoside in UF-resin solution in an open tube allows the water to evaporate causing the condensation of the resin to a cross-linked insoluble polymer. Any glucoside that reacted with the resin will be retained by the insoluble resin. It was found, however, that the glucoside could be quantitatively recovered by extracting the cross-linked resin with cold water. This finding clearly excludes the possibility of bond formation between the glucoside and the resin during the polymerization of the latter.

The nonreactivity of methyl-α-glucopyranoside toward UF-resin strongly suggests that the carbohydrate hydroxyls do not form ether links with the UF-resin during the curing of resin saturated paper. However, it is not precluded that functional groups present in minor amounts in the pulp, such as carboxyl and aldehyde groups, might be reactive and may form occasional chemical links between the resin and the fibers.

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An Electron Microscope Study: Slash Pine and Sweetgum Fibers

K. A. JURBERGS

Electron microscopic techniques have been applied to investigating the structure of pulp fibers from slash pine and sweetgum. The evidence obtained from both wood species indicate that the primary wall is removed during the production of dissolving grade pulps. The surface of the S_1 (outer layer of the secondary wall) layer which covers the fibers consists of two systems of fibrils crossing each other at an angle of about 140°. Two systems of fibrils crossing in wide arcs have also been found on the surface of the pine-bordered pit cavities. The pit annulus appears as a separate structure, possibly consisting of lignin. The torus consists of a circular primary part with secondary thickenings. Several of the perforated pit closing membranes found are believed to belong to the semibordered type from the ray crossfields.

Only during the last decade have the different specimen preparation techniques for the electron microscope been successfully applied to objects of biological origin and detailed information has been forthcoming about the morphology of cellulose fibers. The fine structure of the fibers is not yet completely established and a number of controversial pieces of evidence and interpretations have been published. The present work discusses the evidence obtained from approximately 200 electronmicrographs on the fine structure of selected wood fibers. The results presented may help to clarify certain points on the morphology of cellulose fibers.

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MATERIALS AND METHODS

Wood pulps prepared from slash pine (*Pinus elliottii* Engelm.) and sweetgum (*Liquidambar styraciflua* L.) were used for the present study. The cellulose is possibly only slightly modified and can be expected to resemble closely the native form of cellulose I as found in the wood itself.

The majority of the electronmicrographs were obtained from one-step chromium replicas of the fiber

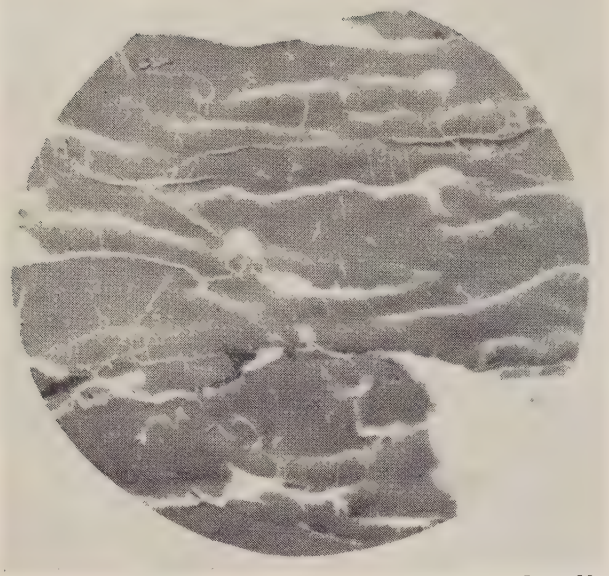

Fig. 1. The surface of an unbleached paper grade sulfate pine fiber. All the electronmicrographs are reproduced at a magnification of 5900×

surfaces. The chromium was evaporated on the fibers at an angle of 45°, stripped by embedding in collodion, and supported by an evaporated carbon membrane. The electron microscope specimen was finally obtained by dissolving the collodion in amyl acetate. It consisted of the chromium replica and the carbon supporting membrane.

A few of the specimens were prepared from polystyrene replicas. Several others were obtained from mechanically dispersed cellulosic material. The very fine, dispersed fiber wall fragments were shadowed with chromium at a low angle of about 20°.

THE SURFACE OF SLASH PINE TRACHEIDS

The surface of an unbleached pine sulfate paper grade pulp fiber is flat and smooth. As seen in Fig. 1, no signs of the characteristic cellulose fibrils can be

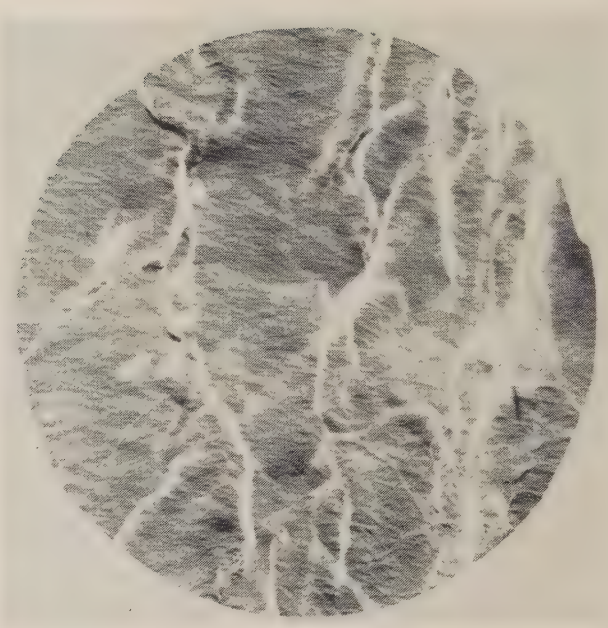

Fig. 3. The surface of a bleached pine sulfite fiber

detected, and the surface appears structureless. With more severe purification conditions, such as used for the manufacture of dissolving grade pulps, more material is removed from the fiber walls. The cellulose fibrils become apparent and their arrangement in the different wall layers can be examined.

The evidence from the present study indicates that the primary wall P is removed from both sulfate and sulfite dissolving grade pulp fibers. Only remains of P covering small areas, or in the form of disarranged bundles of fibrils, can be occasionally found on the fibers. Figure 2 shows such a small area of P on a sulfite pulp fiber.

The surface of a dissolving grade pine pulp fiber is covered exclusively by the outermost layer of the secondary wall S_1. It has been pointed out by Emerton (*1*) and Wardrop (*2*) that the structure of this layer is more complex than the generally accepted present day concept. Evidence obtained from the

Fig. 2. Remains of the primary wall on a bleached pine sulfite fiber

Fig. 4. A bleached pine sulfite summerwood fiber. An exposed area of S_2 is seen in the upper left corner

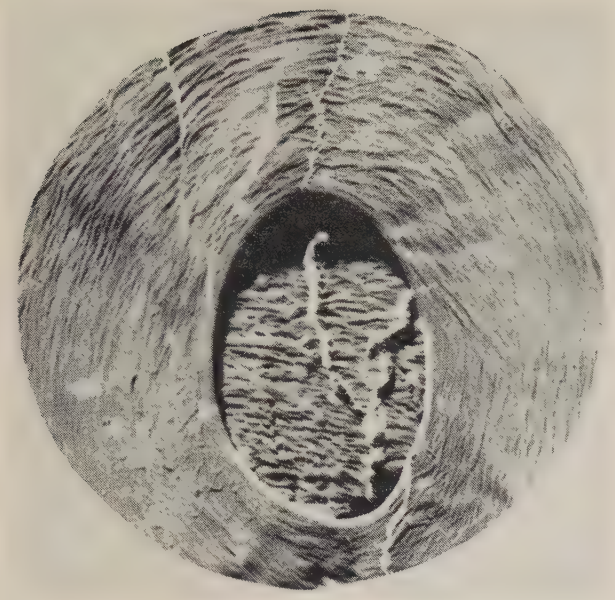

Fig. 5. The S_3 layer from the lumen side observed through the pit aperture. A bleached pine sulfate springwood fiber

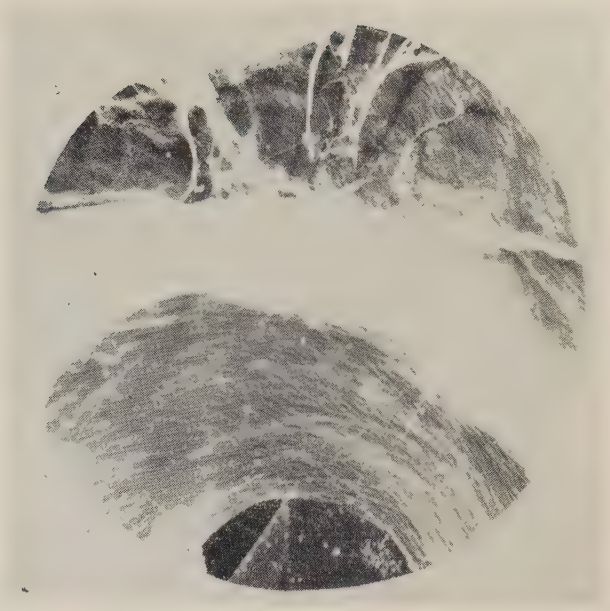

Fig. 7. A pseudoreplica of the annulus. Unbleached pine sulfate fiber

present work confirms observations of the above workers that the outside surface of this layer consists of two systems of spirals of fibrils crossing each other at a wide angle. The surface of a sulfate pulp fiber is covered by the S_1 layer as is shown in Fig. 3. Measurements of the crossing angle have indicated an average value of 140° with only slight variations from area to area of the same fiber or from different fibers. The helix angle (between the tangent of a spiral and the fiber axis) therefore has an average value of 70°. The S_1 layer may be damaged but it was never found completely removed from the fiber surfaces. Contrary to Lange's (*3*) statement, it has been found in our work that two systems of spirals also become apparent during the first phases of swelling of pine fibers. These spirals are visible under the light microscope, have completely different dimensions, and apparently consist of aggregates of fibrils. During the course of the swelling one of the systems soon becomes dominant and the other disappears.

The S_2 layer was exposed in the few cases where the S_1 layer was partially removed. The fibrils in this layer are arranged strictly parallel to each other in rather steep spirals (a helix angle of about 30°). Such an area is seen in the upper left corner of Fig. 4.

Areas of the S_3 layer have occasionally been observed from the lumen side. The opposite wall of the fiber is sometimes replicated through the pit apertures from which the pit closing membrane has been removed. Figure 5 illustrates this point.

THE PITS OF PINE TRACHEIDS

In the dome shaped pit cavities the fibrils lie in broad nonconcentric arcs around the pit apertures. Again two systems of fibrils, crossing each other at approxi-

Fig. 6. A bordered pit of a bleached pine sulfite fiber. Few of the "warts" seen on the walls of the pit cavity

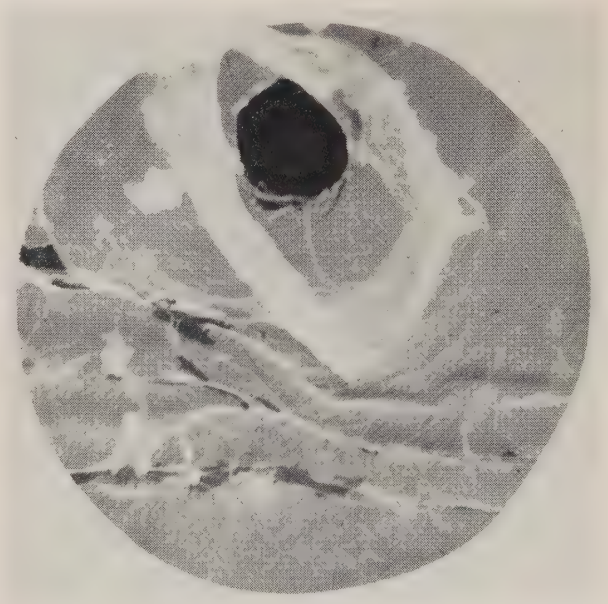

Fig. 8. A pseudoreplica of a dislocated pit annulus. Bleached pine sulfite fiber

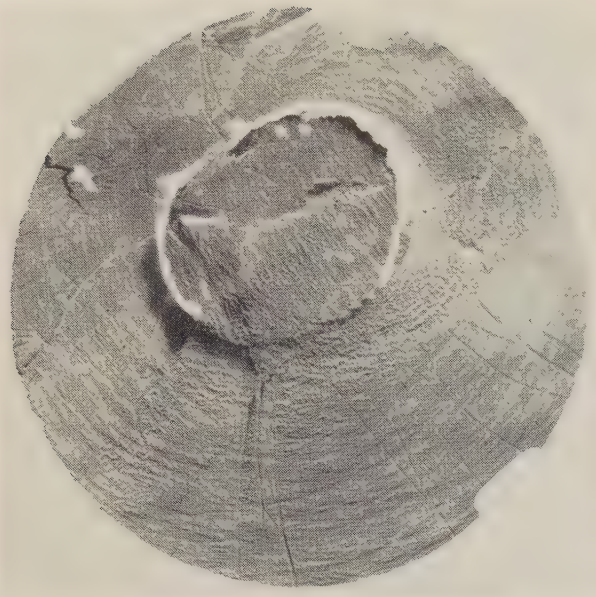

Fig. 9. A pit closing membrane of a bleached pine sulfate springwood fiber

Fig. 11. Part of the pit closing membrane isolated from mechanically dispersed pine chlorite holocellulose

mately the same angle as in the S_1 layer of the fiber wall proper, are apparent. Figure 6 shows the cavity of a bordered pit of a sulfite fiber.

The pit annulus, which originally surrounds the border of the recess of the pit cavity, can be isolated as a separate structural element. On the electgonmicrograph in Fig. 7 the pit annulus is seen as a dark circular ribbon, apparently in its original position around the pit cavity. It can also be found either partially displaced, or hanging loosely over the pit as seen in Fig. 8. It is believed that these structures are pseudoreplicas and the annulus itself is adhering to the replicas of the fiber wall. No fibrillar structure is discernible, and it is possible to speculate that the annulus consists mainly of lignin. It is either a part of the true middle lamella, or more likely a highly lignified structure of the primary wall. As far as the author is aware, its isolation as a separate structure has not been previously reported.

Since the pit closing membranes are parts of the primary wall it is not surprising that in most cases the closing membranes have been removed along with the rest of this layer. According to Bailey (*4*) we can expect to find great variations in the structure of pit closing membranes even among pits from the same tree. One of the very few cases with only partially removed pit closing membrane from a bordered pit of a springwood fiber is shown in Fig. 9. Several of the radial threads and a part of the torus are apparently missing but the overall architecture of the whole closing membrane is indicated. The electronmicrographs show also some nonfibrous material which possibly has solidified on the surface after diffusing from the fiber wall.

Quite frequently pits are found with tori completely closing the pit apertures. The rest of the pit closing membrane is usually missing. Figure 10 illustrates

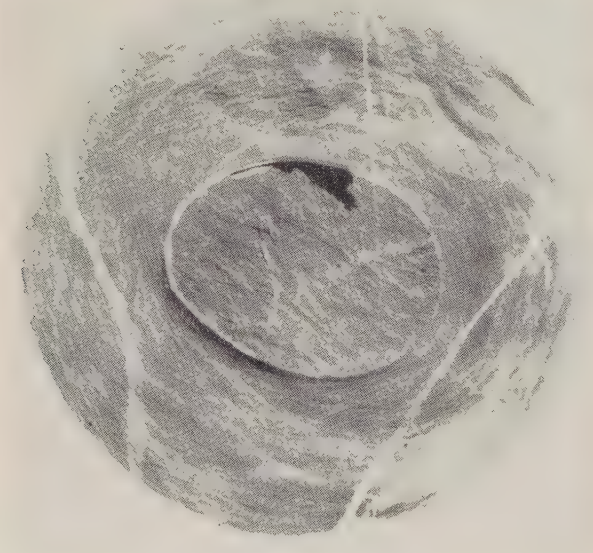

Fig. 10. An aspirated torus glued to the pit aperture. A bleached pine sulfite fiber

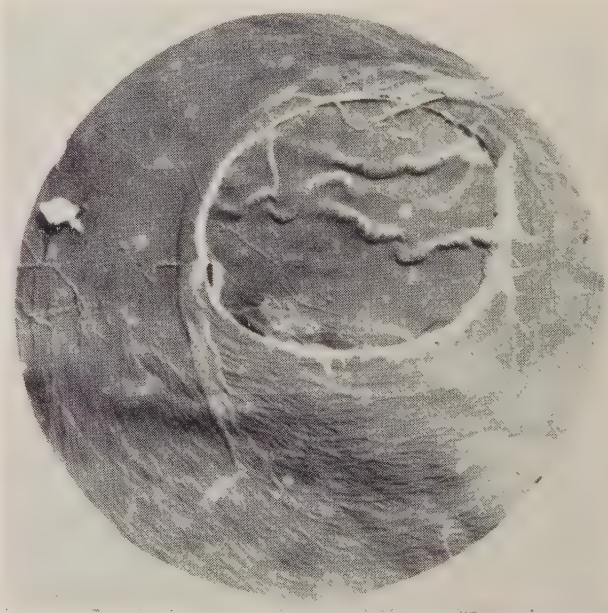

Fig. 12. Torus of a bordered pit on a bleached pine sulfite fiber

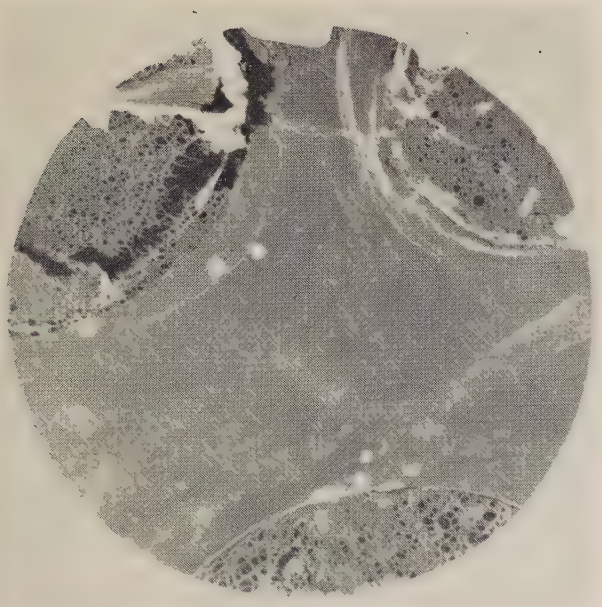

Fig. 13. Perforated pit closing membranes of a bleached pine sulfite fiber

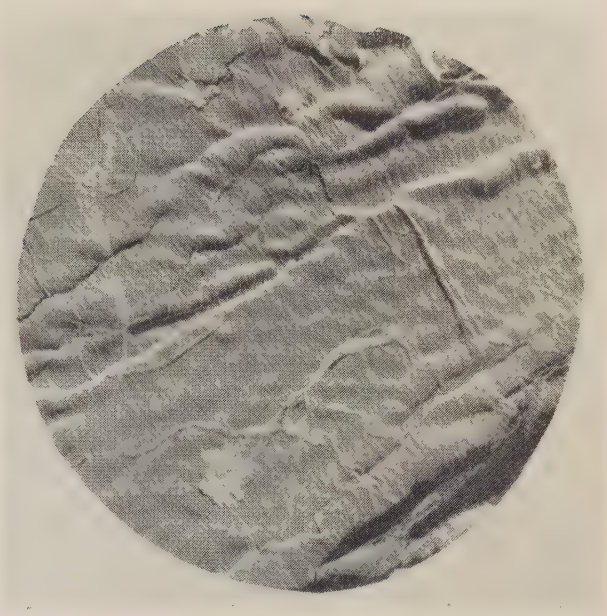

Fig. 15. The surface of a bleached sweetgum fiber tracheid

such a bordered pit, possibly from the heartwood where the tori have been aspirated to one of the pit apertures and have become glued in this position with resins. Such cases have been reported by Wenzl (*5*).

The tori of at least some of the bordered pits appear to consist of two parts. The primary part of the torus is seen in Fig. 11 as a circular bundle of fibrils with some of the radial threads attached to it. This particular structure was isolated from a sample of mechanically dispersed pine chlorite holocellulose. The secondary part of the torus may have been added on each side of the circular bundle of fibrils opposite each of the pit apertures of a pit pair. The tori do not seem to be expressly disk shaped. They are rather flat with occasional transverse rodlike reinforcements. Such reinforcements are depicted on the torus in Fig. 12.

The next two electronmicrographs (Figs. 13 and 14) show pit closing membranes with perforations. The oblong perforations are not uniform in size or shape. Similar perforations have been reported to Stemsrud (*6*) and Liese (*7*). The perforated closing membranes discussed here belong to the semibordered pits from a ray crossing area.

THE STRUCTURE OF SWEETGUM FIBERS

The surface structure of sweetgum fibers is very similar to that of slash pine fibers. The primary wall is removed during pulping from both the fiber tracheids and the vessel segments. Remains of the primary wall only occasionally can be found on the fibers. These remains usually are found in the form of several disarranged individual bundles of fibrils. The surfaces of the fiber tracheids and vessel segments are covered with the S_1 layer. Figures 15 and 16 show typical surface areas of the fiber tracheids, and Fig. 17 that of a vessel segment. Again two systems of fibrils crossing each other are discernible. The small number of measure-

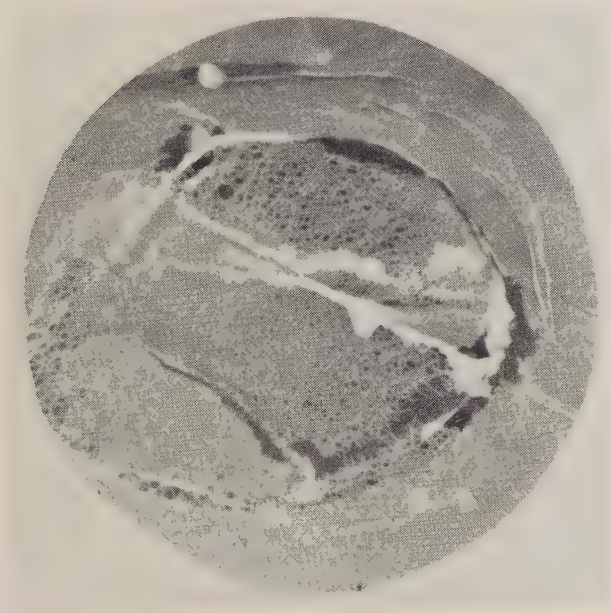

Fig. 14. One of the pits from Fig. 13

Fig. 16. A bleached sweetgum fiber tracheid

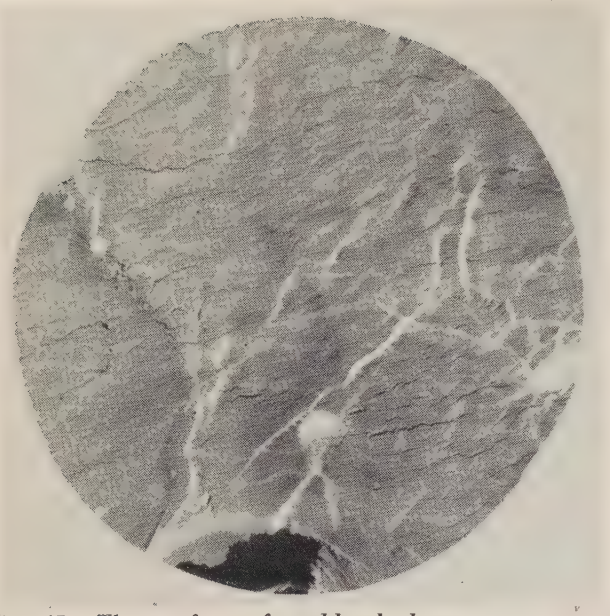

Fig. 17. The surface of a bleached sweetgum vessel segment

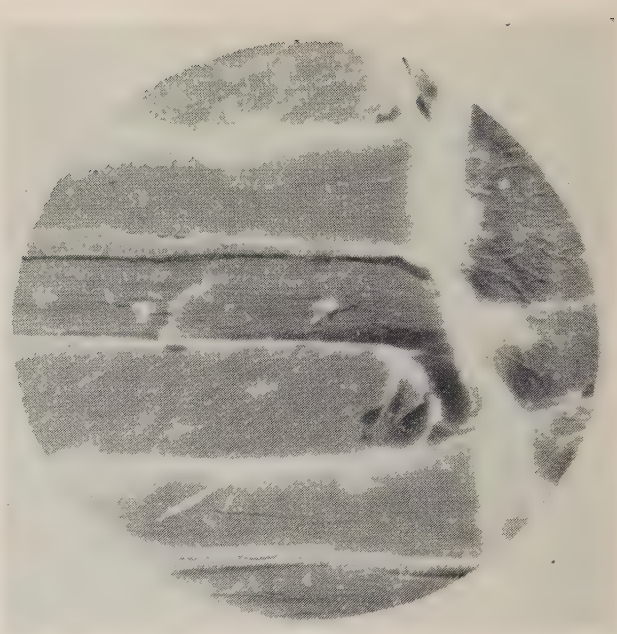

Fig. 19. Linear intervessel pits of a bleached sweetgum vessel segment

ments are not sufficient for accurate determination of the crossing angle for the sweetgum fibers. The few measurements made indicate that the angle for both fiber types (vessel and fiber tracheid) is roughly the same as found for the pine tracheids.

An example of a bordered pit of a fiber tracheid is depicted in Fig. 16. Great variations in size and shape are found among these pits. The pit cavities are much shallower than the corresponding structures on pine tracheids. No evidence of a pit annulus, or any parts of the closing membranes, has been found so far. These structures, if present originally, have been apparently removed during pulping.

Figure 18 shows an oval intervessel pit together with parts of other bordered pits of a vessel segment. In the upper right corner of the micrograph there is a corner of a linear pit originally connecting two vessel segments. A series of these linear intervessel pits is depicted in Fig. 19. In all these cases the pit apertures are closed by pit closing membranes. An electron micrograph from a pseudoreplica of the bars from the scalariform end plate of a vessel segment is shown in Fig. 20.

CONCLUSIONS

Purification processes used for manufacturing of dissolving grade pulps from slash pine and sweetgum wood remove the primary wall from the pulp fibers together with the interfiber bonding materials. The surfaces of the fibers are exclusively covered by the outer layer of the secondary wall S_1. Two systems of fibrils are discernible on the surface of this layer. The fibrils from both systems cross each other at a wide angle. Measurements of this angle have indicated that the helix angle for the S_1 layer of pine tracheids is about 70°. The few measurements of the sweetgum fibers

Fig. 18. Intervessel pit on the surface of a bleached sweetgum vessel segment

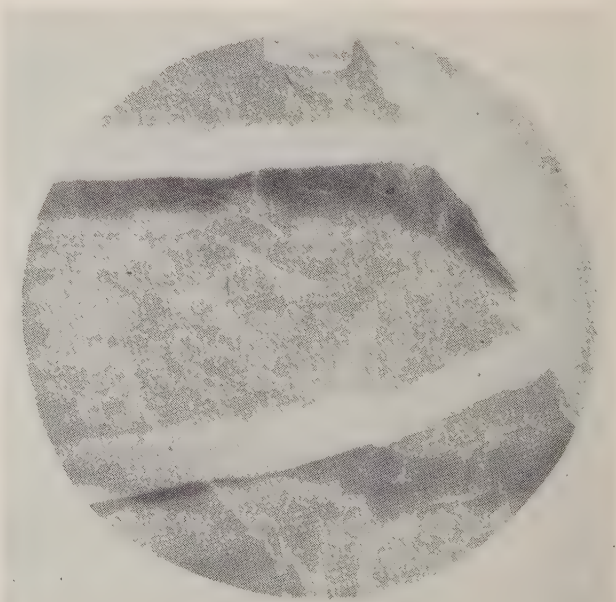

Fig. 20 Pseudoreplica of the bars from a scalariform endplate of a sweetgum vessel segment

indicate only slight deviations, if any, from this value for both the fiber tracheids and vessel segments.

Fibrils in the cavities of the bordered pits of pine fibers are arranged in two systems crossing each other in wide nonconcentric arcs. Measurements of this crossing angle indicate that it is roughly the same as that for the fiber wall proper.

The pit annulus of the bordered pits of pine tracheids is a separate entity consisting of a nonfibrous material, possibly lignin. Similar structures have not been found from pits of the sweetgum fibers.

As a general rule, the pit closing membranes are either partially or completely removed together with the primary wall. A number of the pit apertures are frequently closed by the torus. The evidence obtained indicates that the pit closing membrane of a pine tracheid consists of two parts. The primary part (a part of the primary wall) consists of a centrally located circular bundle of fibrils suspended over the pit apertures by the threadlike radial bundles of fibrils. A secondary thickening of the torus, slightly larger than the pit aperture, develops later on each side of the central circular bundle opposite the two apertures of a pit pair. The torus occasionally closes completely one of the pit apertures and can remain in this position even after the rest of the closing membrane is removed. Perforated pit closing membranes, believed to be connected with the semibordered pits, have been found on several pits of a pine tracheid.

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Chemical Reactivity of Rayon Pulps

Some Aspects of Vapor and Emulsion Xanthation

HIROMICHI HARA, NAOYASU KIMURA, KEINOSUKE NAKAO, and ISAO KIMURA

The behavior of alkali cellulose from rayon pulps during vapor and emulsion xanthation is clarified and some relationships are given between reactivity and the pulp species: sulfite, sulfate, and linter. Although it is well known that the quality of rayon is greatly affected by the pulp materials used, a quantitative relationship has not been established because of the complicated chemical and physical changes which occur in viscose process. Certain aspects of chemical reactivity have been investigated especially the relationship between mercerized and sulfurized reactivity, and the constitution of rayon pulps. Three typical pulps (sulfite, sulfate, and linters) were used in this study. Both vapor and emulsion xanthation were studied to determine the reaction properties of the pulps, and the molecular weight distribution and crystallinity of the original pulps were examined.

EXPERIMENTAL

Pulp Analysis

The analytical characteristics of the pulps are shown in Table I. Of these, linters is the highest-grade pulp for rayon; as can be seen from its alpha-cellulose content, alkali solubility, and copper number value. Except for the fairly large amount of pentosan, sulfate pulp is intermediate in these properties, and sulfite pulp is a general textile-grade pulp.

Determination of Degree of Polymerization (D.P.) Distribution

The distribution of the D.P. was determined by the fractional precipitation method reported by Mitchell (*1*). The viscosity was measured in an Ostwald viscometer at 20 ± 0.50°C. The intrinsic viscosity and D.P. of each fraction were calculated using the following equation:

$$[\eta] = \frac{\eta_{sp.}/c}{1 + k'\eta_{sp.}} \qquad (1)$$

$$D.P. = [\eta]/k_m \qquad (2)$$

where

$$k' = 0.35$$

$$k_m = 11 \times 10^{-4}$$

The pulps were mercerized in 18% NaOH for 1 hr. at 20°C., and pressed to a press ratio of 2.7. The aging conditions of the alkali celluloses are shown in Table II, their average D.P. was controlled at approximately 400.

Determination of Degree of Crystallinity

The aq. HCl hydrolysis method of Philipp and Nelson (*2*), and Nagano (*3*) was used for this determination. The pulp sample was fluffed in an electric blender and hydrolyzed in 3.5 *N* HCl under reflux for various times, up to 6 hr. The hydrolyzed residues were washed with 2 *N* NH_4OH in order to remove humic substances, dried thoroughly, and weighed. The logarithm of the

Table I. Pulp Analysis

Type / *Mark*	*Sulfite pulp* A	*Sulfate pulp* B	*Sulfate pulp* C	*Linter pulp* D
Relative viscosity	4.6	4.3	3.1	4.3
α-Cellulose, %	92.5	94.7	95.2	98.8
β-Cellulose, %	4.9	2.4	3.1	1.2
Pentosan, %	...	2.3	1.1	...
1% NaOH solubility, %	3.1	1.5	2.3	1.0
Alcohol-benzene extn., %	0.11	0.20	0.15	0.08
Copper no.	1.1	0.4	0.5	0.4

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Table II. Aging Condition

	Pulp			
	A	B	C	D
	D.P.			
	900	810	620	790
Aging temp., °C.	29	26	22	26
Aging time, hr.	18	19.5	21.5	18

weight of the hydrolyzed residues was plotted against the time of hydrolysis and the crystalline portion was obtained by extrapolating to zero beyond the turning point.

Method of Vapor Sulfurization. Aged alkali cellulose was sulfurized using 40% CS_2 in a 1000-ml. glass vessel in which the pressure had been previously lowered to 160 mm. Hg. During the reaction, the flask was revolved at 14 r.p.m. in a water bath at 28°C.

The cellulose xanthate was dissolved in NaOH to give a viscose containing 6% cellulose and 5% caustic soda. Combined sulfur was determined by Bredée's method.

To evaluate the reactivity, the equation developed by Sakurada was used (*4*):

$$Sx = kt^m \qquad (3)$$

where
Sx = combined CS_2, %
k and m = constants
t = reaction time, hr.

The reaction velocity was obtained from equation (*3*) as follows:

$$dSx/dt = kmt^{m-1} \qquad (4)$$

The logarithmic form of equation (3) is:

$$\log Sx = \log k + m \log t \qquad (5)$$

From the log-log curve of Sx and t, the values of k and m were obtained.

Method of Emulsion Xanthation. The Bartunek method was used for the determination of mercerization and sulfurization resistance (M.R. and S.R.) (*5*).*

Lateral Order Distribution from Emulsion Xanthation

Migita and Nakao (*6*) have reported a new method for the determination of lateral order Q in which the M.R. method of Bartunek is modified. In the determination of M.R., the alkali concentrations were varied from 6 to 12% and the per cent solubility at every alkali concentration was obtained by subtracting M.R.% from 100. The lateral order distribution curve was obtained from the differential solubility curve as a function of alkali concentration.

To eliminate the D.P. effect, methanolyzed pulp, pulp which had been subjected to methanolysis, as well as the original pulp was studied. The methanolysis solution was composed of 5 volumes of 11 N HCl and 95 volumes of methanol. The crushed pulp sample was methanolyzed under reflux for 4 hr. The residue was washed several times with methanol and dried at 50°C.

Determination of Solubility and Sulfidity

The solubility and sulfidity experiments followed the same method as for M.R. and S.R., but after reaction, the undissolved portions were separated immediately from the solution. M.R. and S.R. were calculated according to Bartunek. The determination of the gamma-number of the dissolved portions was performed according to Bredée.

* In ref. (*5*) M.R. = M.W., and S.R. = S.W.

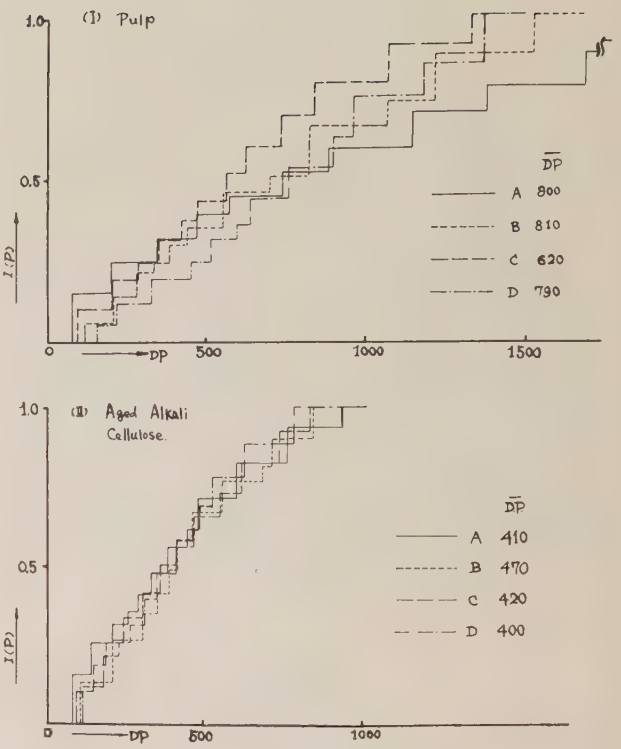


Fig. 1. Degree of polymerization distribution curves of pulp and aged alkali cellulose

RESULTS AND DISCUSSION

Properties of the Original Pulps

Remarkable differences were observed in D.P.

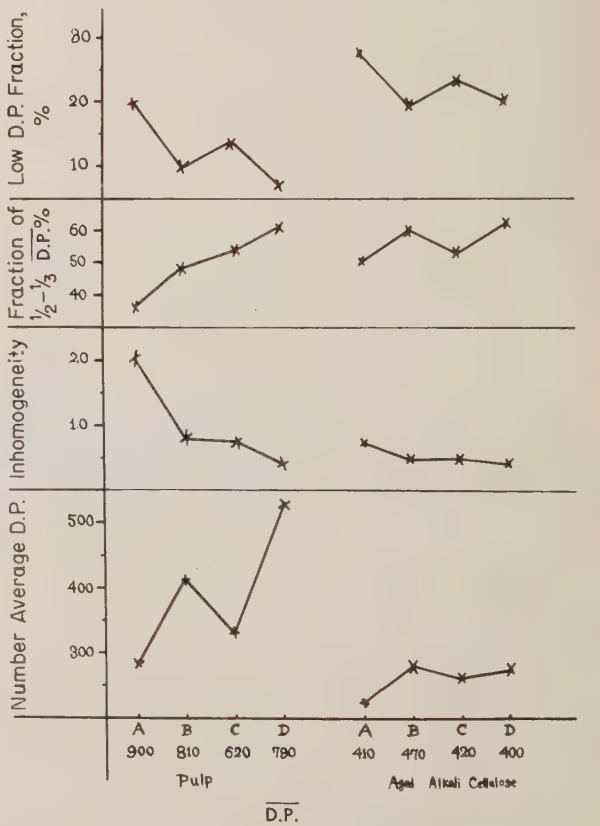


Fig. 2. Degree of polymerization distribution characteristics of pulp and aged alkali cellulose

Table III

	Pulp A	B	C	D
Co, %	88.7	89.0	89.3	92.0
$Ka \times 10^3$, hr.$^{-1}$	16.8	19.3	13.6	16.1
$Kc \times 10^3$, hr.$^{-1}$	5.8	5.9	8.2	5.6
Turning point position on time-axis, hr.	3.4	3.1	3.6	2.7

Co = Crystalline region.
Ka = Reaction velocity constant of crystalline region.
Kc = Reaction velocity constant of amorphous region.

distribution among the original pulps, as shown in Figs. 1 and 2. Linters showed the least inhomogeneity in D.P. distribution, sulfate pulp is intermediate, and sulfite pulp has the widest distribution.

Differences in the D.P. distribution of pulps decrease during aging of the alkali cellulose. Although cellulose molecules become uniform in length on aging, aged alkali cellulose, however, still retains the D.P. properties of the pulp and the relation distribution is the same as that for the original pulps.

Table III shows that the pulps are approximately 90% crystalline; linters show the highest degree of crystallinity followed by sulfate and sulfite pulps. The turning point on the hydrolysis curve for the linters is situated to the left and is high compared to the others (Fig. 3). These facts indicate rapid removal of the amorphous region in linters.

Vapor Xanthation

The xanthation curve for sulfite pulp is parabolic

Table IV

	Pulp A	B	C	D
k	23.71	23.67	22.50	16.22
m	0.125	0.103	0.110	0.221
km	2.964	2.425	2.472	3.570

and shows a maximum sulfidity, while the curve for linters ascends gradually with reaction time (Fig. 4). The curve for sulfate pulp is similar to that for sulfite initially but its slope does not decrease until after 6 hr.

By employing Sakurada's equation, the sulfidity curves for linters and sulfate pulp become linear (Fig. 5). The break in the plot for sulfite pulp shows a drop in sulfidity owing to autodecomposition. As

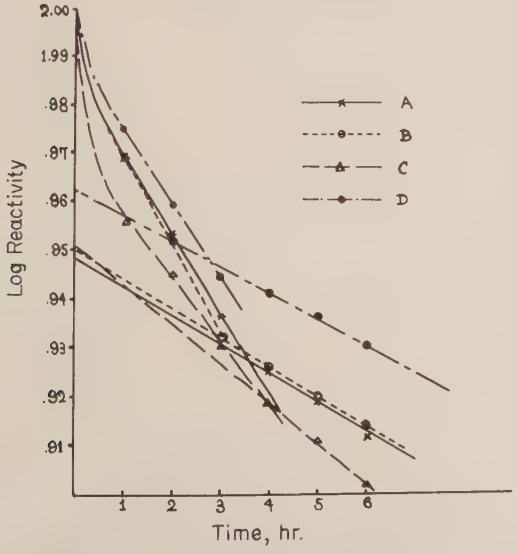


Fig. 3. Hydrolysis of pulp with 3.5N HCl at 100°C

Table V. Mercerization Resistance

	Pulp A	B	C	D
D.P.	900	810	620	790
M.R., %	29.5	70.5	77.4	95.2

shown in Table IV, linter pulp, which has a small accessible portion and good internal order, shows the lowest value of k and the highest value of m. On the contrary, wood pulp shows a high value of k and low value of m, and the difference between sulfite pulp and sulfate pulp is little observed.

This may point out that k indicates the amount of CS_2 which is absorbed by the accessible part of alkali cellulose at the beginning of xanthation, and m is a parameter expressing CS_2 affinity for alkali cellulose during the xanthation. It may then be assumed that k is a function of the accessibility of cellulose and m is related to the homogeneity of the molecular structure of cellulose.

Emulsion Xanthation

Mercerization Resistance. In the M.R. experiments, linters show the highest resistance and sulfite the lowest (Table V). In the process of emulsion xanthation the mercerized portions of the pulp are sulfurized, and these portions should be swollen or dissolved by alkaline solution. However, because of the low alkali concentration there are no mercerized or partially mercerized

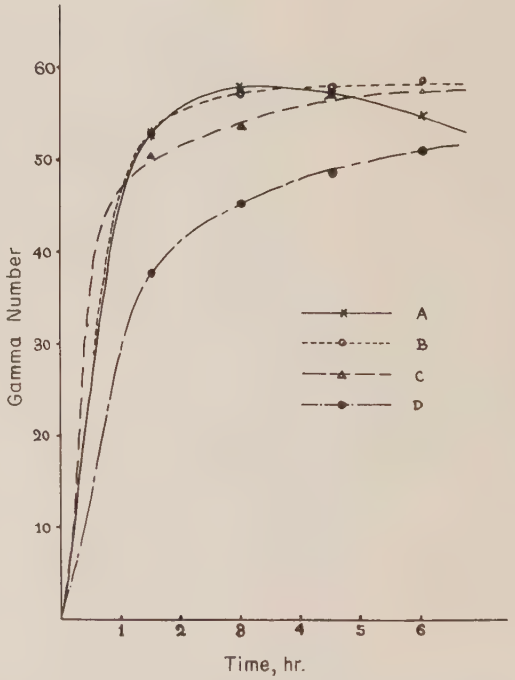


Fig. 4. Sulfurization of pulp with 40% CS_2/cellulose at 28°C

portions in the laterally bonded regions of cellulose molecules.

The lateral order distribution curves shown in Fig. 6 (I) were obtained from the solubilities in M.R. experiments. The curve for sulfite pulp has a low-order maximum, that for linters is of a high order, and for sulfate pulp it is intermediate.

The lateral order distribution curves of pulps freed from the D.P. effect by methanolysis are shifted to the

Table VI. Amount of Gelatinized Portion (II) during M.R. Determination, %

A	B[a]	B[b]	D
32.2	24.2	22.2	4.1

[a] Unextracted pulp.
[b] Pulp extracted with alcohol-benzene mixed soln.

left, but the relative order of the maxima does not change from that of the original pulps. Therefore, there are remarkable differences in the lateral cohesion of the cellulose molecules in these three pulps. The cohesion is strongest in linters and weakest in sulfite pulp.

Pulp which has a lower solubility shows also a small amount of gelatinized material during the dissolving process, which is indirectly obtained by washing the reacted cellulose with dilute NaOH solution. Moreover, the dissolved portion of this pulp has a high sulfidity (Figs. 7, 8, and Table VI). Since the lateral bonding forces of these pulps are stronger, the pulps

Table VII. Sulfurization Resistance

		A	B	C	D
Pulp	$\overline{D.P.}$	900	810	620	790
	S.R., %	39.4	45.8	35.4	21.5
	S.R./$\overline{D.P.}$ × 100	4.4	5.7	5.7	2.7
Aged alkali cellulose	$\overline{D.P.}$	410	470	420	400
	S.R., %	16.2	25.6	14.6	8.3
	S.R./$\overline{D.P.}$ × 100	4.2	5.4	3.5	2.1

are mercerized to a lesser degree. Consequently, M.R. expresses the state of lateral bonding; i.e., the lateral cohesion of the cellulose molecules.

Correlation of Mercerization Resistance and Vapor Xanthation. From these facts, it may be concluded that a pulp which shows the higher M.R. value, has the smaller xanthation reactivity in the vapor phase in spite of uniform gamma-number distribution. In vapor xanthation, however, these pulps show smaller differences in lateral order mercerization because they have lost their highly ordered regions (*7*). Among wood pulps, the degree of lateral order is affected by the pulping process (Fig. 6) but in vapor xanthation the sulfidity, after mercerization, is not affected (Figs. 4 and 5).

Since pulps which show a high M.R. value have also a high lateral order and crystallinity, as reported by Philipp (*8*), it is necessary to break more hydrogen bonds and hence to expend more energy to cause the transition from alkali cellulose to cellulose xanthate

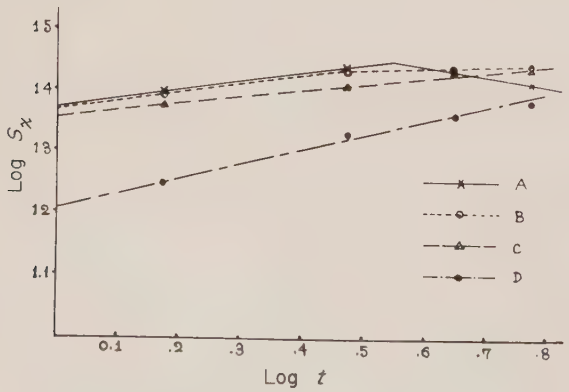


Fig. 5. Log-log form of Fig. 4 from equation (5)

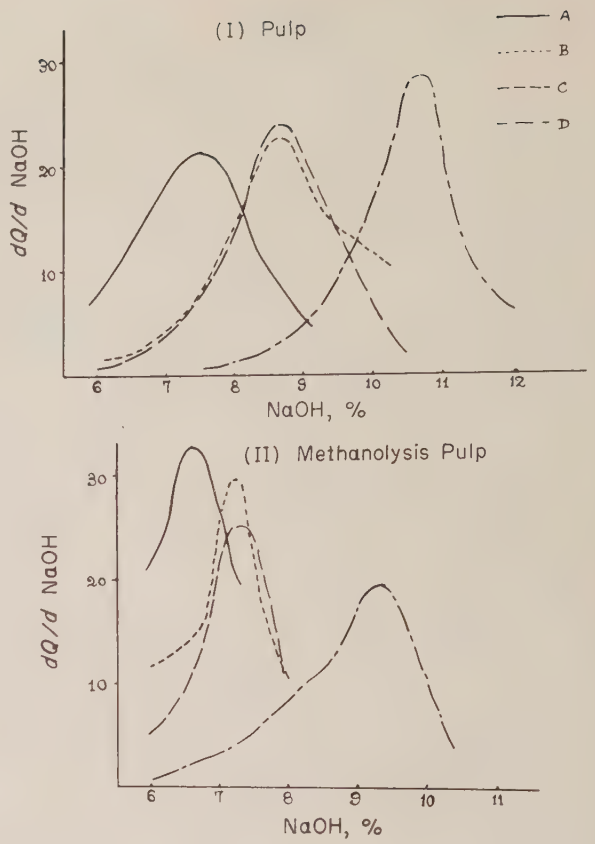


Fig. 6. Lateral order distribution curves at emulsion xanthation

by vapor xanthation. Such pulps therefore have a lower reactivity in vapor phase xanthation.

Sulfurization Resistance. In the S.R. experiments, linters show the lowest value (Table VII). In the determination of S.R., the sample was mercerized completely by 18% NaOH solution. The sample which shows low swelling properties and low solubility, owing to its structure or high D.P., gives the highest S.R. value because of an insufficient amount of combined CS_2. If, because of the uneven structure of the

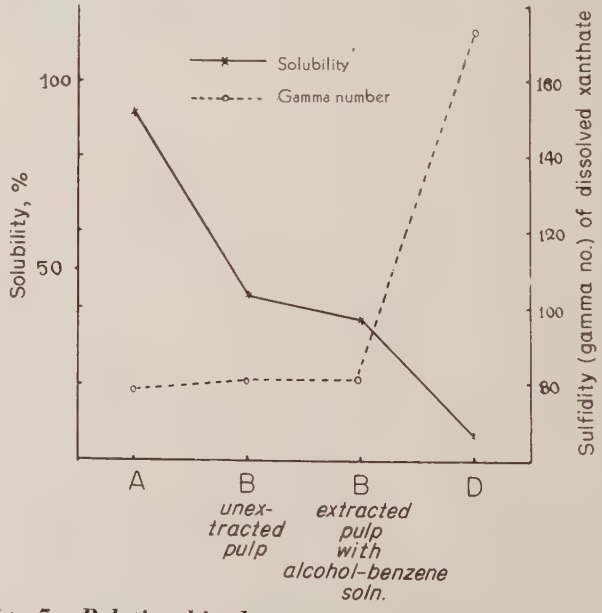
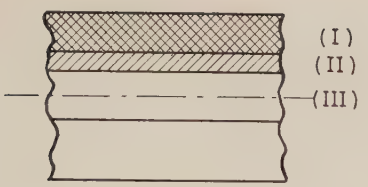


Fig. 7. Relationship between solubility and sulfidity during M.R. determination



(I) Dissolved portion
(II) Gelatinized portion
(III) Undissolved portion

Fig. 8. A model of dissolving state of cellulose at emulsion xanthation

sample, consumption of CS_2 is incomplete, the distribution of sulfidity in such a sample becomes inhomogeneous and low solubility results. That is to say, S.R. is influenced by the structural characteristics and D.P. of the cellulose. To eliminate the D.P. effect, we have tried to use a numerical factor (S.R./D.P. $\times$ 100). As a result, sulfate pulp shows the highest value and linters the lowest. It should be noted that the former has a low swellability and solubility, while the latter has a relatively uniform structure. The reason for the deviation of the sulfate pulp is not evident, but it may depend upon the pulping process (*9*). In the case of aged alkali cellulose, linters also show the lowest value. The values of the numerical factors for alkali cellulose are a little lower than those for the original pulps because the former become more reactive during the aging process.

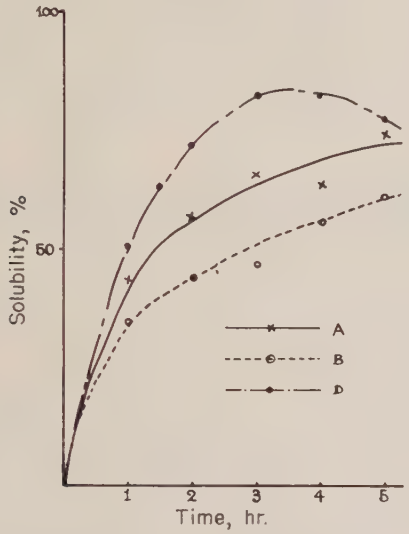


Fig. 9. Variation in solubility with reaction time S.R. determination

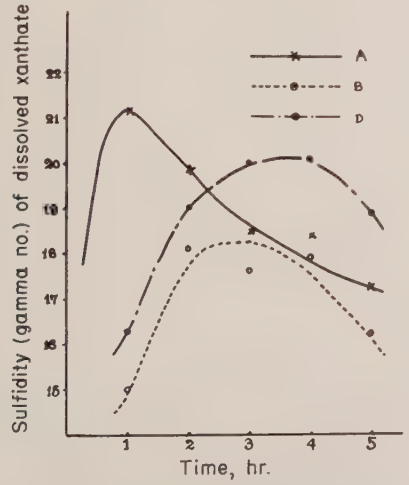
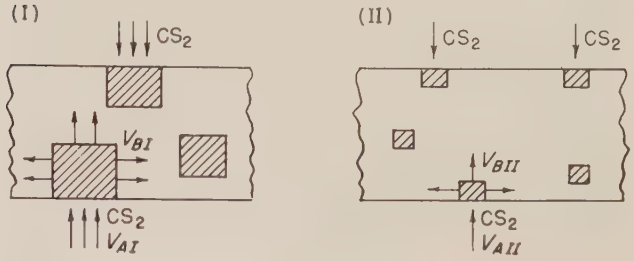


Fig. 10. Variation in sulfidity with reaction time at S.R. determination



Accessible region

Va: Absorption velocity of CS_2 by alkali cellulose.
Vb: Diffusion velocity of absorbed CS_2 to unreacted portions.
Vc: Autodecomposition velocity of Na-cellulose xanthate.

Fig. 11. A model of the reaction mechanism of emulsion xanthation

Figures 9 and 10 show the dissolving behavior of emulsion xanthation products in S.R. experiments. The characteristic curves in Fig. 10 can be explained by a schematic diagram, as shown in Fig. 11. We now assume that three reaction velocities, *Va*, *Vb*, and *Vc*, are the factors which control the solubility and sulfidity. These are related as follows:

	Solubility of dissolved xanthate	*Sulfidity of dissloved xanthate*
Va—Absorption velocity of CS_2 by alkali cellulose	+	+
Vb—Diffusion velocity of absorbed CS_2 to unreacted portion of cellulose	+	−
Vc—Autodecomposition velocity of Na-cellulose xanthate	−	−

It is considered that the reaction velocity is proportional to the accessible surface, so that the pulp absorbs CS_2 selectively in the accessible regions at the beginning of the reaction (Fig. 11). This mechanism corresponds to the behavior of the sulfite and sulfate pulps. Pulp with a small accessible surface behaves as shown in Fig. 11 (II). The absorption (*Va*) takes place uniformly on many small accessible parts, and new accessible surfaces are created by the dissolving out of xanthate. Thus, *Va* increases with reaction time, a behavior that corresponds to that of linters (Fig. 12).

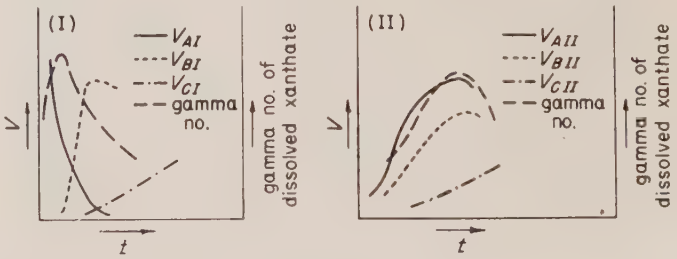


Fig. 12. Reaction model curves during emulsion xanthation

Correlation of Sulfurization Resistance and Vapor Xanthation. From the foregoing results, it is evident

that there is no relationship between S.R. and the rate of vapor xanthation, i.e., the sulfidity curve for emulsion xanthation (Figs. 9 and 10) does not correspond to that of vapor xanthation (Figs. 4 and 5). The peculiarity of emulsion xanthation is the production of mercerization and xanthation simultaneously. The receptivity of different celluloses to CS_2, especially in their accessible regions, can be predicted from their dissolving behavior in S.R. determinations because of the presence of small amounts of CS_2. The role of the accessible portions of cellulose in S.R. is similar to that in vapor xanthation, and the initial high gamma-value in Fig. 10 fits the k value in Fig. 5. Thus, it is clear that uniform xanthation can take place in a pulp having relatively high M.R. and low S.R. values.

SUMMARY

1. We have shown that sulfite pulp has the most disordered fine structure, as shown by such properties as D.P. distribution, crystallinity, and lateral order distribution. Linters show the opposite behavior, and sulfate pulps are intermediate.

2. The pulp having the highest degree of crystallinity and lateral order has the highest M.R. value, while its reaction velocity in vapor xanthation is small; linters is a typical example.

3. There is no relation between S.R. value and the rate of vapor xanthation.

4. In vapor xanthation uniform xanthation is achieved by using pulp which has a high M.R. and low S.R. value.

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Chemimechanical Pulps from Various Softwoods and Hardwoods

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The results of experiments on the making of high-yield chemimechanical* pulps from hardwoods and softwoods by several cooking techniques are discussed in this report. In one method of neutral sulfite chemimechanical pulping a conventional procedure was followed, while in another method a hot-liquor impregnation procedure was followed. Another chemimechanical pulp was made using a two-stage acid alkaline process. Aspen, western hemlock, and a mixture of hardwoods were pulped by the conventional neutral sulfite chemimechanical method at yields ranging from 87 to 92%. Balsam fir and western hemlock were impregnated and cooked with a preheated neutral sulfite liquor to yields ranging from 90 to 97%. Western hemlock was also pulped by the two-stage acid-alkaline chemimechanical process. The effect of varying the pH of the cooking liquors by the use of various buffers was determined. The various types of chemimechanical pulp are compared in regard to properties and energy consumptions. Selected pulps were bleached and used in papermaking experiments. Newsprint papers were made by substituting high-yield hardwood neutral sulfite chemimechanical pulps for softwood sulfite pulp. Magazine-grade papers were made by substituting high-yield softwood neutral sulfite chemimechanical pulps for part of both the softwood groundwood and sulfite components.

The possibility of producing useful pulps in the yield range of about 90% or more by refining chips softened by a mild chemical treatment has attractive possibilities for better wood utilization. Pulps of this type made from many hardwood species, which produce groundwood pulp too weak for general use, have properties approaching those of softwood-groundwood pulps. Pulps made from some softwoods in a similar way generally have the high bulk and opacity characteristics of groundwood pulp and have, in addition, higher strengths that justify their consideration as partial substitutes for softwood chemical pulps in some blends.

G. H. Chidester, Chief, Division of Pulp and Paper, J. F. Laundrie, Chemical Engineer, E. L. Keller, Chemical Engineer, Forest Products Laboratory, maintained at Madison, Wis., in cooperation with the University of Wisconsin, Forest Service, U. S. Department of Agriculture.

* Chemimechanical pulps are defined in this report as pulps produced from wood in any form or shape by using a mild chemical treatment in which there is no major change in the lignin, followed by a mechanical treatment to separate the fibers. Usually these pulps fall within the yield range of 80 to 95%.

CHEMIMECHANICAL PULPING

Two cooking techniques were used to make the high-yield neutral sulfite chemimechanical pulps. In one method a conventional procedure was followed; that is, presteaming, liquor addition, gradual temperature rise, and a time at maximum temperature followed by blowdown. To obtain high yields of pulp by this method, less chemical was dissolved in the cooking liquor than for normal semichemical pulping, and the maximum cooking temperature was reduced to between 130 and 140°C. These temperatures were comparable to those recommended for securing bright pulps by the chemigroundwood process.

Hot-liquor impregnation cooks were made by heating the neutral sulfite liquor in a pressure reservoir connected to the digester, before introducing it to the chips. During the impregnation periods, which lasted from 10 to 40 min., a hydrostatic pressure of 100 p.s.i.g. was maintained in the digester. Preheating the impregnating solution to temperatures between 170

and 190°C. produced equilibrium temperatures in the digester of from 125 to 135°C. At the end of the impregnation, the liquor was blown back into the reservoir, where it was measured and sampled for analysis.

To make the two-stage acid-alkaline chemimechanical pulp, the chips were presteamed, followed by an evacuation of the digester to about 20 in. Hg. A sodium bisulfite liquor at room temperature was then added, followed by the injection of liquor sulfur dioxide. The temperature was raised to 114°C., where it was held for 35 min. After blowdown, the treated chips were passed through a screw press and then returned to the digester, where they were boiled at atmospheric pressure for 3 hr. in a solution of sodium bicarbonate.

Aspen, western hemlock, and a mixture of hardwoods consisting of 50% red maple and 10% each of red oak, white oak, yellow birch, ash, and elm were pulped by the conventional-type process in a yield range of about 87 to 92%. Hot-impregnation cooks were made on balsam fir and western hemlock at yields ranging from about 90 to 97%. Western hemlock was the only wood pulped by the two-stage acid-alkaline chemimechanical process at a yield of about 81%.

Conventional-Type Cooks

Effect of Cooking and Species. There was very little difference between the amounts of sodium sulfite required for cooking the aspen and the hardwood mixture by the conventional procedure, although the mixture did require a longer cooking time. Table I shows that the amount of cooking, as measured by the yield, is critical, since opacity, as well as strength, is sensitive in this respect. The loss in opacity on cooking to lower yields is accompanied by a reduction in the amount of energy required for refining, as well as by a marked increase in pulp strength. Considering pulp brightness, strength, and energy consumption in refining, aspen is indicated to be better than the mixture of denser hardwoods. The density of the aspen test sheets was considerably greater than that of the sheets made of the mixed hardwoods, which suggests that the strength difference could be reduced by increased wet pressing of the latter. The lower density of the mixed hardwood pulp sheets is probably due to the oaks, since these species are known to give bulky pulps. Because of the differences in brightness between the

Table I. Properties of Unbleached Chemimechanical Pulps from Various Hardwoods and Softwoods, Pulped by Different Processes

Pulping process	[*Yield,*[a] %	*Energy consumed,*[b] *hp.-days*	*Freeness (C.S.), ml.*	*Burst factor*	*Tear factor*	*Breaking length, m.*	*Density, g. per cc.*	*Opacity,*[c] %	*Brightness (G.E. equivalent), %*	*Screen classification — On 28-mesh, %*	*Retained between 28- and 48-mesh, %*	*Retained between 48- and 100-mesh, %*	*Retained between 100- and 200-mesh, %*	*Passing 200-mesh, %*	*Fiber length, index*
Aspen															
Neutral sulfite	88	69	180	23	52	4250	0.61	88	49	..	..	..	..	..	...
Neutral sulfite	92	99	180	12	33	2960	0.46	94	51	..	..	..	..	..	...
Cold soda	88	33	250	36	63	6350	0.62	75	..	..	..	..	..	..	...
Cold soda	92	36	335	19	60	3600	0.61	..	54	24	29	25	8	14	0.153
Chemigroundwood	89[d]	38	185	54	70	8580	0.75	75	40	22	33	19	7	19	0.159
Chemigroundwood	91[d]	41	245	31	78	5710	0.60	..	55	16	42	16	8	18	0.133
Groundwood	97[e]	99	70	13	44	3050	0.40	94	59	3	19	27	20	31	0.082
Groundwood[f,g]	97[e]	..	60	11	39	2400	0.38	..	60	8	16	22	16	38	0.083
Mixed Hardwoods[h]															
Neutral sulfite	87	95	180	20	55	3440	0.46	95	41	..	..	..	..	..	...
Neutral sulfite	92	104	180	6	26	1530	0.34	98	38	..	..	..	..	..	...
Cold soda	90	..	180	26	53	4400	0.57	..	42	..	..	..	..	..	...
Cold soda	92	..	180	14	42	3040	0.44	..	44	..	..	..	..	..	...
Bisulfite	90	143	170	8	27	2030	0.36	98	38	2	11	34	15	38	0.073
Balsam Fir															
Neutral sulfite[i]	91	78	325	26	92	4640	0.44	85	51	..	..	..	..	..	...
Neutral sulfite[i]	92	97	260	31	94	5260	0.47	87	50	..	..	..	..	..	...
Neutral sulfite[i]	95	71	320	22	85	2940	0.43	83	53	..	..	..	..	..	...
Cold soda[j]	..	49	245	17	49	3080	0.44	86	47	..	..	..	..	..	...
Bisulfite	90	57	380	26	113	4230	0.34	88	46	60	18	11	4	7	0.266
Chemigroundwood	96[d]	82	45	26	84	4850	0.44	86	56	37	14	14	11	24	0.112
Groundwood	97[e]	56	113	15	51	3100	0.40	..	59	16	17	24	14	29	0.093
Western Hemlock															
Neutral sulfite[i]	87	73	310	35	95	5520	0.49	83	42	57	13	6	3	21	0.138
Neutral sulfite[i]	93	77	260	24	101	4060	0.37	91	47	37	25	15	7	16	0.151
Two-stage acid-alkaline[k]	81	41	400	25	103	4150	0.50	68	53	53	17	9	4	17	0.156
Groundwood	97[e]	..	100	13	54	2575	0.43	95	54	8	21	23	16	32	0.083
Sulfite[f]	46[l]	..	620	49	93	7380	0.73	67	42	56	17	13	3	11	0.203
White Spruce															
Sulfite[f]	48[l]	..	450	39	100	7250	0.72	78	53	..	..	..	..	..	...

[a] Moisture-free weight basis.
[b] Per ton of airdry pulp.
[c] Sheet weight 37 lb. per ream, 500 sheets 25 × 40 in.
[d] Based on change in specific gravity of wood before and after treatment.
[e] Assumed water soluble loss of 3%.
[f] Commercial pulp.
[g] Made from a mixture of 25% aspen, 65% spruce, and 10% balsam.
[h] Mixture consisted of 50% red maple, and 10% each of red oak, white oak, yellow birch, ash, and elm.
[i] Hot liquor impregnation treatment with sodium bicarbonate buffer.
[j] Continuous.
[k] Chips first cooked in an acid sulfite solution and then in an alkaline solution before mechanical fiberization.
[l] Yield assumed for bleachable pulp.

Table II. One-Stage Bleaching of Mixed Hardwood Neutral Sulfite Chemimechanical Pulp at 87.0% Yield

Bleaching chemical — Formula	Amount	Temperature, °C.	Consistency, %	Duration, min.	pH	Brightness (G.E. equivalent), %
$Ca(OCl)_2$[a]	10.0	36	10	60	11.6–9.6	74.0
$Ca(OCl)_2$[a]	8.0	36	10	30	11.8–10.4	69.5
$Ca(OCl)_2$[a]	8.0	37	10	45	12.2–12.0	69.6
$Na_2S_2O_4$[b]	1.5	60	3	30	6.5–6.7	60.8
$Na_2S_2O_4$[b]	1.5	60	3	60	6.9	61.6

[a] Calcium hypochlorite buffered with 4% sodium silicate solution and 4% sodium hydroxide. Percentages are in terms of available chlorine.
[b] Sodium hydrosulfite stabilized with 0.5% trisodium salt of ethylene diamine tetraacetic acid.

aspen and mixed hardwood pulps, conclusions cannot be drawn as to comparative opacity.

Bleaching. The mixed hardwood chemimechanical pulp was bleached in order to estimate the chemical requirement and also the opacity at a brightness suitable for use in printing papers. The pulp responded readily to moderate amounts of bleach in a single-stage application of either calcium hypochlorite or sodium hydrosulfite. As shown in Table II, applying 8% available chlorine as calcium hypochlorite for 30 min. at a temperature of 36°C. and a consistency of 10% gave a brightness of 69.5%. Using these same conditions and extending the duration 15 min., with a slight rise in pH, gave approximately the same final brightness. Using a consistency of 3% and a temperature of 60°C., the application of 1.5% sodium hydrosulfite for 30 and 60 min. gave brightness values of 60.8 and 61.6%, respectively.

Table III. Effect of Bleaching a Mixed Hardwood Neutral Sulfite Chemimechanical Pulp at 87% Yield

Properties	Unbleached pulp	Bleached pulp
Freeness (C.S.), ml.	140	155
Burst factor	21.5	20.4
Tear factor	49.2	51.6
Breaking length, m.	3840	3750
Density, g. per cc.	0.50	0.52
Opacity (37-lb. ream), %	91.8	81.8
Brightness (G.E. equivalent), %	46.7	69.6

It is shown in Table III that the strength of the pulp was not significantly changed by bleaching to a brightness of 69.6%, although the opacity dropped 10 percentage points to an unsatisfactory level of 81.8%.

Impregnation-Type Cooks

From the standpoint of pulp color and brightness, hot-impregnation sodium sulfite treatments, using sodium bisulfite as the buffer, were found to be more satisfactory than those buffered with either sodium carbonate or bicarbonate in producing high-yield

Table IV. Effect of pH in Producing High-Yield Neutral Sulfite Chemimechanical Pulps from Balsam Fir[a]

Impregnating liquor — Buffer	pH	Na_2SO_3 used,[b] %	Yield,[b] %	Brightness (G.E. equivalent), %
Na_2CO_3	10.4	9.0	90.3	47.6
$NaHCO_3$	8.8	13.2	92.1	49.7
$NaHCO_3$	8.3	11.7	91.0	51.2
$NaHSO_3$	7.6	10.0	94.8	52.5

[a] Constant impregnating conditions: 10 min. at 135°C. with a liquor having a Na_2SO_3 concentration of 60 g.p.l.
[b] Moisture-free wood basis.

Table V. Substitution of Aspen High-Yield Neutral Sulfite Chemimechanical Pulp for Softwood Groundwood in Newsprint Paper

Properties	Experimental[a] paper	Commercial[b] paper
Ream weight (500 sheets, 25 × 40 in.), lb.	39.4	37.2
Thickness, mils	3.9	2.9
Density, g. per cc.	0.56	0.72
Bursting strength, pt. per lb.	0.25	0.20
Tearing resistance, g. per lb.	0.74	0.65
Tensile strength, lb. per in. width	10.2	7.2
Folding endurance, double folds	5	2
Opacity, %	87.1	90.2
Brightness (G.E. equivalent), %	62.3	60.5

[a] Furnish consisting of 20% southern pine sulfate pulp and 80% aspen neutral sulfite chemimechanical pulp (91% yield). Headbox freeness—305 ml.
[b] Standard furnish of softwood sulfite and groundwood pulps.

pulps from balsam fir. As shown in Table IV, lowering the pH from 10.4 to 7.6 during a series of 10-min. treatments at 135°C. increased the brightness of the pulp from 47.6 to 52.5%, and decreased the yellow cast noticeably without significantly affecting pulp strength. The highest yield of pulp was produced when the pH value was the lowest, and the lowest yield of pulp was produced when the pH value was highest.

PAPERMAKING

Substitution of High-Yield Neutral Sulfite Chemimechanical Hardwood Pulp for Softwood Groundwood

An experimental newsprint paper was made on a 13-in. fourdrinier paper machine from a furnish consisting of 20% southern pine sulfate pulp beaten to a freeness of 320 ml. (C.S.), and 80% high-yield (91%) neutral sulfite chemimechanical aspen pulp processed to a freeness of 440 ml. The experimental paper (Table V) had more strength and a higher brightness when compared with a sample of commercial Canadian newsprint. The opacity of the experimental newsprint is somewhat lower than the Canadian newsprint, although Laboratory experiments have shown that disk-mill refining of the neutral sulfite chemimechanical pulp to a lower freeness or the addition of a small

Table VI. Substitution of Balsam Fir High-Yield Neutral Sulfite Chemimechanical Pulp for Part of Both Groundwood and Sulfite Pulps in Magazine-Base Papers

	Machine run no. 4943	4948	4950
Pulp furnish:			
Bleached spruce-aspen groundwood, %	60	50	60
Bleached western hemlock sulfite, %	40	25	25
Bleached balsam fir neutral sulfite chemimechanical (92.9 per cent yield), %	...	25	15
Headbox freeness (C.S.), ml.	130	145	140
Ream weight (500 sheets, 25 × 40 in.), lb	31.8	33.2	32.0
Density, g. per cc.	0.61	0.54	0.54
Thickness, mils	2.9	3.4	3.3
Bursting strength, pt. per lb.	0.21	0.24	0.24
Average tearing resistance, g. per lb.	0.75	0.72	0.70
Average folding endurance, double folds	3	3	2
Average tensile strength, lb. per in. width	6.1	6.2	6.4
Opacity, %	83.2	84.6	84.7
Brightness (G.E. equivalent), %	78.4	77.0	76.7

amount of clay could have increased this value. Refining to a lower freeness also might have increased the density of the experimental newsprint.

Substitution of High-Yield Neutral Sulfite Chemimechanical Softwood Pulp for Both Groundwood and Sulfite Pulps

The substitution of high-yield balsam fir pulp, in the amount of 25% for part of both the groundwood and the sulfite components of a magazine stock, decreased the density somewhat and slightly increased the opacity of the papers without significantly affecting the strength properties (Table VI). Other papers made from 60% bleached groundwood, 25% bleached sulfite, and 15% of the bleached balsam fir neutral sulfite chemimechanical pulp compared very closely with the

Table VII. Substitution of Western Hemlock High-Yield Chemimechanical Pulps for Sulfite Pulp in Newsprint Papers

	Machine run no.			
	5331	*5333*	*5332*	*5334*
Western hemlock pulp furnish:				
Commercial groundwood, %	75	75	75	75
Commercial sulfite, %	25	...	...	...
Experimental neutral sulfite chemimechanical:				
93 per cent yield, %	...	25	...	...
87 per cent yield, %	...	...	25	...
Bleached chemimechanical[a] (81% yield), %	...	...	...	25
Headbox freeness (C.S.), ml.	200	200	170	200
Ream weight (500 sheets, 25 × 40 in.), lb.	37.9	38.1	37.0	39.4
Density, g. per cc.	0.70	0.57	0.62	0.61
Thickness, mils	3.0	3.7	3.3	3.6
Bursting strength, pt. per lb.	0.19	0.12	0.15	0.11
Average tearing resistance, g. per lb.	0.66	0.44	0.51	0.48
Average folding endurance, double folds	6	3	3	2
Average tensile strength, lb. per in. width	7.9	5.8	6.3	5.8
Opacity, %	89.3	93.2	89.2	89.5
Brightness (G.E. equivalent), %	51.7	50.1	55.5	53.3

[a] Made by a two-stage, acid-alkaline process.

experimental reference standard paper, which consisted of a 60–40 mixture of groundwood and sulfite pulps.

Experimental newsprint papers made by substituting western hemlock two-stage acid-alkaline and neutral sulfite chemimechanical pulps at two yield levels for all of the sulfite components were not as strong as the reference standard paper, which was made from a pulp furnish consisting of 75% western hemlock groundwood and 25% western hemlock sulfite pulp (Table VII). The papers containing the lower yield neutral sulfite (86.9%) and the two-stage acid-alkaline chemimechanical pulps were somewhat stronger than the one containing the higher yield pulp (93.4%), although the opacity of the paper containing the higher yield pulp was distinctly higher than either the reference standard paper or the experimental papers containing the lower yield neutral sulfite and the two-stage acid-alkaline chemimechanical pulps. The brightness values of the reference standard paper and the paper containing the higher yield neutral sulfite chemimechanical pulp were about the same, while the brightness value of the papers containing the lower yield neutral sulfite and the two-stage acid-alkaline chemimechanical pulps were about 5 and 3 percentage points higher, respectively.

COMPARISON OF VARIOUS KINDS OF HIGH-YIELD PULPS

Included in Table I are data for typical high-yield pulps, ranging from 88 to 97% in yield, made by different processes from aspen and a mixture of denser hardwoods, and also from balsam fir and western hemlock. It is recognized that, for any given process, yield, and species, pulps varying in properties are obtainable because of variations in fiber processing, grinder operation, wood quality, and other factors. Therefore, while comparison of single pulps selected as representative types may indicate fairly definite trends, the comparisons must not be considered as strict generalizations or final answers. The comparisons are made on the unbleached pulp basis. If these points are kept in mind, the following comparison of different kinds of high-yield pulps may afford some indication of general trends.

Opacity

High opacity was obtained in aspen and mixed hardwood pulps produced in yields above 90% by the neutral sulfite, and bisulfite chemimechanical, and the groundwood processes. Aspen cold soda and chemigroundwood pulps of 88 and 89% yield, respectively, were relatively low in this important property.

Regardless of yield, the balsam fir and western hemlock neutral sulfite, cold soda, and bisulfite chemimechanical pulps were low in opacity. The two-stage acid-alkaline chemimechanical pulp also had a low opacity, about the same as the commercial western hemlock sulfite pulp. The only exception to this was a western hemlock neutral sulfite chemimechanical pulp of 93% yield, which had an opacity of 91%. Western hemlock groundwood pulp had an opacity of 95% and, though not available, the opacity of the balsam fir groundwood could be expected to be similarly high.

The opacity of pulp is influenced by its brightness and also by the fiber characteristics and size distribution. Pulps in this yield range that have high opacity are often low in brightness.

Brightness

Pulps made in this yield range by mild chemical treatment are generally low in brightness regardless of the method of preparation. At yields above 90%, the brightness of aspen neutral sulfite, cold soda, and chemigroundwood chemimechanical pulps are fairly comparable to the brightness of unbleached groundwood pulps from aspen and the softwoods. The brightness of all the aspen pulps below 90% yield is less than that of the groundwood pulps. The brightness of the mixed hardwood pulps, regardless of yield or process, is also less than the brightness of the aspen and softwood-groundwood pulps.

Of the softwood pulps, only the balsam fir chemigroundwood pulp was relatively close to the softwood-groundwood pulps in brightness. Those softwood high-yield pulps that could, because of their strength, be considered as partial substitutes for unbleached sulfite pulp were as good as, or better than, the spruce

and western hemlock sulfite pulps in brightness. If the unbleached pulps are restricted in usefulness because of their low brightness, they may be readily bleached by the processes used for bleaching groundwood. However, bleaching invariably reduces opacity, so the pulps with high initial opacity are most likely to have the advantage for bleaching treatments. Although the yellowness of the various pulps produced was not measured quantitatively, it was observed visually that the cold soda pulps, even after bleaching, had more of a yellow cast than any of the other pulps.

Strength

The association of higher strength with lower yield was noticeable in even the narrow range of 88 to 92% yield for both the neutral sulfite and cold soda chemimechanical pulps. At the same yield levels, the hardwood cold soda pulps had better strength than the hardwood neutral sulfite and hardwood bisulfite pulps. The aspen chemigroundwood pulps were stronger than the aspen neutral sulfite and cold soda pulps at the same yield and freeness levels. Both the aspen and mixed hardwood cold soda pulps at 92% yield were comparable to softwood ground woods in strength.

The balsam fir bisulfite, the neutral sulfite, and the chemigroundwood chemimechanical pulps made at 90, 91, and 97% yields, respectively, were comparable in strength. However, the freeness of the chemigroundwood was very much lower than the other two. The balsam fir cold soda pulp was similar to balsam fir groundwood pulp in strength.

The most noticeably comparable strength value between certain of the softwood high-yield pulps and the acid sulfite pulps made from the respective species was their tear factor. The tearing strength of the balsam fir bisulfite pulp was higher than that of the spruce sulfite pulp, and the neutral sulfite pulps (at 91 and 92% yield) were almost as high. Similarly comparable tear values are noted for the western hemlock neutral sulfite, two-stage acid alkaline, and sulfite pulps.

Fiber-Size Distribution

The physical character, the chemical composition, and the size distribution of the fibers of which a pulp is composed influence its strength, opacity, freeness, and other properties. The screen classification of a pulp gives an idea of the amounts of the fibers and particles of different lengths or sizes, independent of their physical and chemical characteristics. A few screen classification tests are shown in Table I.

Groundwood pulps generally contain large amounts of material that will pass a 200-mesh screen, and consequently their average fiber length, as indicated by the fiber-length index, is lower than most other high-yield pulps. Pulps in this yield range containing the higher amounts of fines are often higher in opacity and bulkiness of the sheet. For example, in Table I a mixed-hardwood bisulfite chemimechanical pulp, an aspen groundwood pulp, and a western hemlock groundwood pulp have similar screen fractionation values—relatively high amounts of fines and low amounts of long fibers—and are also high in opacity and low in density. The extent to which the fines contribute to these two properties, as well as the strength, depends on whether they are flourlike or are hydrated, and the degree of hydration. Flourlike fines are more likely to give high opacity and bulk than hydrated fines. This may explain why a balsam fir bisulfite pulp and a western hemlock neutral sulfite pulp, which have screen classifications similar to that of a western hemlock sulfite pulp, are higher than the sulfite pulp in both opacity and bulk, but not as good in bursting and tensile strengths.

The stronger high-yield pulps generally contain more long material and have higher fiber-length indexes than the weaker ones.

Density

The aspen neutral sulfite, cold soda, and chemigroundwood chemimechanical pulps all had density values much higher than either the aspen or softwood groundwoods. The only exception to this was a neutral sulfite pulp made at 92% yield, which had a density value only slightly higher than the groundwoods.

It appears that, to produce pulps by the neutral sulfite process from the mixed hardwoods that have density values comparable to those of the groundwoods, the yield must be near 90%, whereas pulps made by the cold soda process must be greater than 92% in yield. A pulp made from the mixed hardwoods by the bisulfite process at 90% yield had a density value actually less than those of the groundwoods.

The softwood chemimechanical pulps, regardless of process or yield, all had density values similar to those of the groundwoods.

Freeness

Hardwood pulps produced by mild chemical treatments with strength and fiber-distribution properties satisfactory for groundwood pulp substitution are generally much freer than the groundwood pulps. On the other hand, if these pulps are strong enough for substitution for softwood sulfite pulp, they are usually lower in freeness than the sulfite pulp.

Energy Consumption

Most of the chemimechanical pulps can be prepared for groundwood pulp substitution with about the same or even less energy consumption than that required for making groundwood pulp. With respect to aspen, the neutral sulfite pulps appear, in Table I, to require somewhat more energy than the cold soda and chemigroundwood pulps at the same yield or the same freeness levels. Energy consumptions for refining the softwood pulps are generally moderate regardless of process.

CONCLUSIONS

Pulps in this high-yield range can be made by several processes with properties varying with the woods used and the conditions used in their preparation.

Their value lies in the fact that in some instances the hardwood pulps can be substituted for softwood pulps, and in other instances the high-yield pulps can be substituted for low-yield pulps.

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ASSOCIATION NEWS AND EVENTS

National Conferences

1960

Fifteenth Engineering Conference, Oct. 24–28, 1960, Robert Meyer Hotel, Jacksonville, Fla.

1961

46th Annual Meeting, Feb. 19–23, 1961, Commodore Hotel, New York, N. Y.
Twelfth Coating Conference, May 22–24, 1961, Statler-Hilton Hotel, Buffalo, N. Y.
Twelfth Testing Conference, Aug. 15–18, 1961, Queen Elizabeth Hotel, Montreal, Que.
Eleventh Corrugated Containers Conference, Sept. 11–13, 1961, St. Francis Hotel, San Francisco, Calif.
Fourth Mechanical Pulping Conference, Sept. 19–21, 1961, Edgewater Beach Hotel, Chicago, Ill.
Symposium on Formation and Structure of Paper, Sept. 25–29, 1961, B.P.B.M.A., Oxford, England.
Sixteenth Plastics-Paper Conference, Oct. 9–11, 1961, French Lick Sheraton Hotel, French Lick, Ind.
Sixteenth Engineering Conference, Oct. 15–19, 1961, Shoreham Hotel, Washington, D. C.
Fifteenth Alkaline Pulping Conference, Nov. 1–3, 1961, Rice Hotel, Houston, Tex.

1962

47th Annual Meeting, Feb. 18–22, 1962, Commodore Hotel, New York, N. Y.
Thirteenth Coating Conference, May 14–16, 1962, Netherland-Hilton Hotel, Cincinnati, Ohio.
Thirteenth Testing Conference, Sept. 18–20, 1962, Benjamin Franklin Hotel, Philadelphia, Pa.
Seventeenth Engineering Conference, Oct. 14–19, 1962, Queen Elizabeth Hotel, Montreal, Que.
Twelfth Corrugated Containers Conference, Oct. 23–26, 1962, Statler-Hilton Hotel, Boston, Mass.
Sixteenth Alkaline Pulping Conference, 1962, Savannah, Ga.

1963

Fourteenth Coating Conference, May 19–22, 1963, French Lick Sheraton Hotel, French Lick, Ind.
Thirteenth Corrugated Containers Conference, Oct. 22–25, 1963, Statler-Hilton Hotel, Detroit, Mich.
Eighteenth Engineering Conference, 1963, Chicago, Ill.

Report of the Nominating Committee

The Nominating Committee has presented the following slate of candidates for officers and Executive Committee members of the Association:

President (1 year): Harold M. Annis, Oxford Paper Co., New York, N. Y.
Vice-President (1 year): John C. Wollwage, Kimberly-Clark Corp., Neenah, Wis.
Executive Committee Members (3 years):
William H. Aiken, Personal Products Corp., Milltown, N. J.
Paul W. Bartholomew, Minnesota & Ontario Paper Co., International Falls, Minn.
Glenn C. Kimble, Union Bag-Camp Paper Corp., Savannah, Ga.
W. P. Lawrence, Champion Paper & Fibre Co., Hamilton, Ohio

Mail ballots will be submitted to active and corporate members of the Association prior to the 1961 Annual Meeting.

According to Article VII, Section 3 of the Association Constitution other nominations, if signed by at least 25 voting members, may be submitted to the Executive Secretary of the Association in writing on or before November 20. If each nomination so made is accepted in writing by the nominee, the name shall be placed on the official ballot.

Members of the Nominating Committee are Edwin H. Olmstead, Eaton-Dikeman Co., Mt. Holly Springs, Pa., chairman; Robert R. Fuller, Gulf States Paper Corp., Tuscaloosa, Ala.; Paul M. Goodloe, Brown Co., Berlin, N. H.; Walter F. Holzer, Crown Zellerbach Corp., San Francisco, Calif.; and Roy P. Whitney, The Institute of Paper Chemistry, Appleton, Wis.

Welcome Mexican Technical Association

A new technical association of the pulp and paper industry in Mexico was formally constituted on Wednesday, August 17, with 90 charter members. It has been named Asociacion Mexicana de Tecnicos de la Industria de la Celulosa y del Papel.

The officers elected were as follows:

President: Adalberto Tirado Arroyave (Loreto and Pena Pobre)
Vice-President: Sergio Vadillo (San Rafael)
Secretary-Treasurer: Aage M. Leth (Celulosa Chihuahua)
1st Vocal: O. Lara Poire (El Fenix)
2nd Vocal: C. Gonzalez (Kimberly-Clark)
Directors:
Jose Sana (San Rafael)
Ricardo Millares (Papel Satinado)
Antonio Hinojosa (Camera del Papel)
Dante Cusi (Industrial San Cristobal)
Hans Lenz (Loreto y Pena Pobre)
Rafael Rojas (Labs Nacionales de Fomento Industrial)

Considerable interest was shown by a large number of persons in the industry. Committees were appointed to get the work of the association under way and to plan for the first meeting which is to be held within a few months.

The new organization has an excellent opportunity to offer a service to the pulp and paper industry in the Spanish-speaking countries. Effort will be made to translate into Spanish technical articles, testing procedures, and information sheets from other languages.

4th Mechanical Pulping Conference (Preliminary Release)

The 4th International Mechanical Pulping Conference is scheduled for Chicago in 1961 and will be held at the Edgewater Beach Hotel, September 19–21. The conference is sponsored jointly by the Technical Association of the American Pulp and Paper Industry and the Technical Section, Canadian Pulp and Paper Association, as were the previous ones held in Poland Springs, Me., and Quebec City, Que. The program being planned will emphasize advances in the technology of mechanical pulping.

The sponsoring groups of this conference are the Mechanical Pulping Committees of each association whose chairmen are, respectively, S. R. Parsons, Consolidated Water Power and Paper Co., Wisconsin Rapids, Wis., and I. Mc-

Arthur, Canadian International Paper Co., Ltd., Montreal, Que.

W. H. de Montmorency, Pulp and Paper Research Institute of Canada, Montreal, Que., is the general chairman of the conference.

14th Alkaline Pulping Conference

The TAPPI 14th Alkaline Pulping Conference was held August 22–24, 1960, at the Multnomah Hotel in Portland, Ore.

This was the first time that the Alkaline Pulping Conference was held in the Pacific Northwest, and as a consequence the Conference was scheduled for the summer to provide vacation opportunities for those planning to attend the Conference. The attendance was excellent, there being 387 men and 97 wives in attendance.

The general conference chairman was D. Wetherhorn, technical director for Continental Can Co., Port Wentworth, Ga., who is also chairman of the Alkaline Pulping Committee.

P. H. West, Pulp Mill Superintendent for Thilmany Pulp and Paper Co., Kaukauna, Wis., was technical program chairman, and he is also vice-chairman of the Alkaline Pulping Committee.

The Sulfite Pulping Committee, recently formed by the merger of the Semichemical Pulping Committee and the Acid Pulping Committee, sponsored one of the four technical sessions of the conference. P. J. Frost, Project Engineer of Crown-Zellerbach Central Research and Development Laboratories, Camas, Wash., was technical program chairman for the Sulfite Pulping Committee session.

J. N. McGovern, Parsons and Whittemore Inc., New York, N. Y., is chairman of the Pulp Manufacture Division of which both the Alkaline Pulping Committee and the Sulfite Pulping Committee are a part.

A. H. Wickett, Superintendent, Weyerhaeuser Co., Longview, Wash., was chairman of the Conference Arrangements Committee. His committee was organized as follows:

Conference Arrangements:
L. M. Shanaman, Pennsalt Chemicals Corp., Tacoma, Wash., assisted by L. S. Dieffenbach, Hercules Powder Co., Inc., Portland, Ore.

Hospitality:
F. Lemma, Northwest Copper Co., Portland Ore., assisted by H. Martin, Hercules Powder Co., Inc., Portland, Ore., and M. J. Maguire, Mfg. Repr., Portland, Ore.

Sulfite Pulping Committee: P. E. Trout, chairman, Waldorf Paper Products Co., St. Paul, Minn.; P. J. Frost, vice-chairman, Crown-Zellerbach Corp., Camas, Wash.; S. R. Parsons, secretary, Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

Registration:
R. D. Baer, Huyck Felt Co., Portland, Ore., and J. M. McCullough, Pennsalt Chemicals Corp., Portland, Ore.

Housing:
J. L. McClintock, Weyerhaeuser Timber Co., Longview, Wash., assisted by G. Petrie, Black-Clawson Co., Portland, Ore.

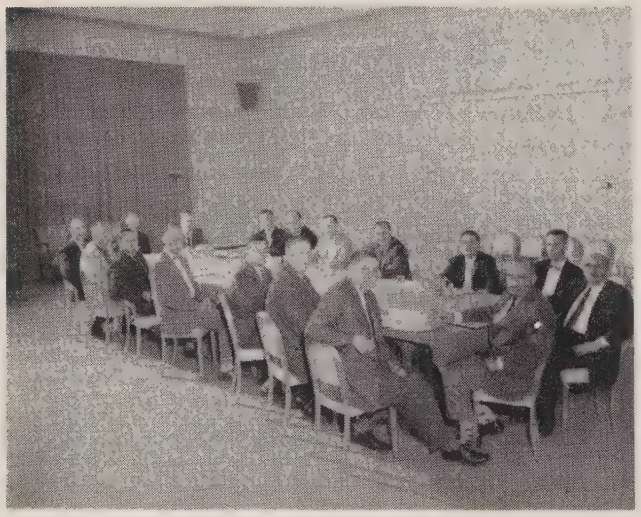
Printing:
E. G. Drew, Drew Engineering Co., Portland Ore., assisted by R. Smythe, Ray Smythe Co., Portland, Ore.

Transportation:
D. B. Armstrong, R. T. Vanderbilt Co., Portland, Ore., assisted by F. T. King, Asten-Hill Mfg. Co., Portland, Ore.

Mill Visits:
R. R. Morris, Crown-Zellerbach Corp., Camas, Wash., assisted by J. M. Wilcox, Electric Steel Foundry Co., Portland, Ore., and L. M. Shanaman, Pennsalt Chemicals Corp., Tacoma, Wash.

Ladies' Program:
F. Caskey, Morden Machines Co., Portland, Ore. assisted by H. B. Peterson, Hercules Powder Co., Inc., Portland, Ore.

As always at the Alkaline Pulping Conferences, the technical sessions were well attended with much discussion of the



Alkaline Pulping Committee: D. Wetherhorn, chairman, Continental Can Co., Port Wentworth, Ga.; P. H. West, vice-chairman, Thilmany Pulp & Paper Co., Kaukauna, Wis.; R. R. Fuller, secretary, Gulf States Paper Corp., Tuscaloosa, Ala.

Alkaline Pulping Session I, left to right: D. Wetherhorn, Continental Can Co., Port Wentworth, Ga.; E. F. Thode, The Institute of Paper Chemistry, Appleton, Wis.; C. W. Rothrock, St. Regis Paper Co., Pensacola, Fla.; R. H. Rasch, Hammermill Paper Co., Erie Pa.; L. A. Carlsmith, Improved Machinery, Inc., Nashua, N. H.; R. T. Mashburn, L. A. Carlsmith, Improved Machinery, Hercules Powder Co., Kalamazoo, Mich.

From the Paper Research Laboratory of The New Jersey Zinc Company
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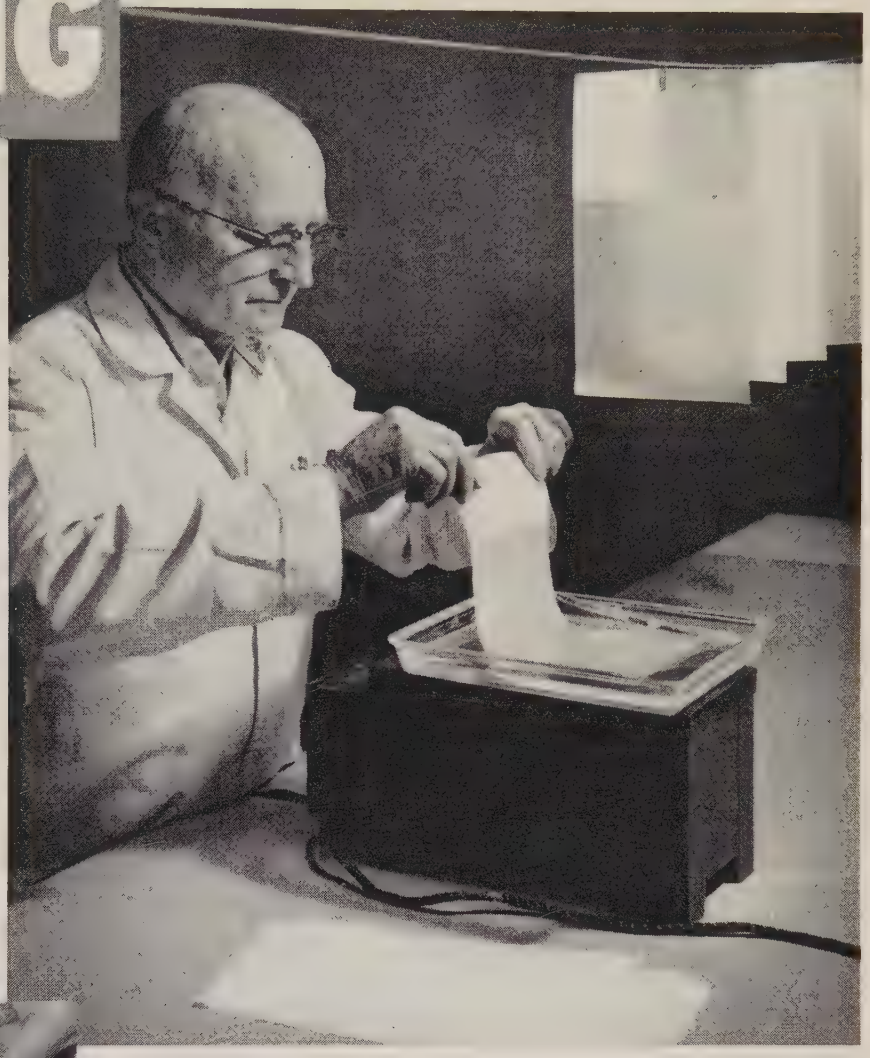
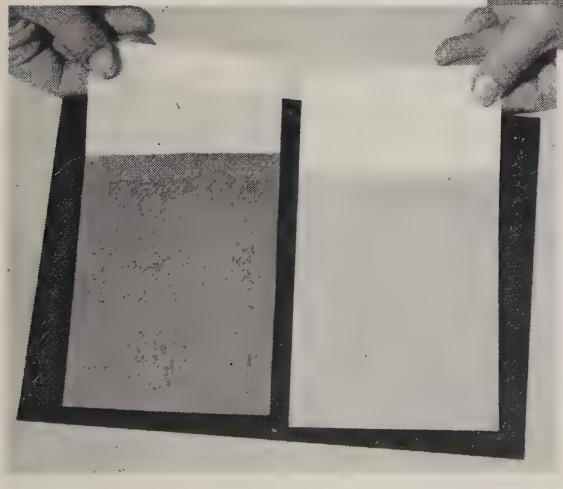


Pigmented and unpigmented handsheets are dipped in molten wax and evaluated for opacity, brightness and other properties essential to modern high grade papers for use in food packaging.



Comprehensive studies on the effect of titanium dioxide on opacity and brightness of waxed papers for food packages are an important phase of the research activities at the paper laboratory of The New Jersey Zinc Company.

Such investigations involve the preparation of handsheets in various weights—pigmented and unpigmented—the application of commercial wax coatings, and extensive evaluations for opacity and brightness.

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Alkaline Pulping Session II, left to right: K. M. Shold, Crown-Zellerbach Corp., Port Townsend, Wash.; V. W. Bacon, Northwest Pulp & Paper Association, Tacoma, Wash.; C. E. Rogers, Babcock & Wilcox Co., New York, N. Y.; H. P. Markant, Babcock & Wilcox Co., Alliance, Ohio; T. C. Payne, International Paper Co., Moss Point, Miss.; B. W. Smith, International Paper Co., Moss Point, Miss.; J. W. Klein, Longview Fibre Co., Longview, Wash.; H. S. Fisher, Pennsalt Chemicals Corp., Tacoma, Wash.

Alkaline Pulping Session III, left to right: E. S. Rice, moderator, St. Regis Paper Co., Jacksonville, Fla.; F. L. Jones, Container Corp. of America, Fernandina Beach, Fla.; B. P. Ellen, Continental Can Co., Savannah, Ga.; J. S. Hart, St. Lawrence Paper Co., Montreal, Que.; L. E. Goodwin, St. Regis Paper Co., Jacksonville, Fla.

presented papers occurring. Abstracts of the papers presented at this conference are appended.

An outstanding feature of the Conference was a speech by Derrick B. Currie, vice-president, Weyerhaeuser Co., Longview, Wash. The subject of Mr. Currie's talk was "Some Thoughts on Leadership." Among the many provocative thoughts which Mr. Currie developed in substantial depth were these: (1) there is no "leadership" type person; (2) leadership is a quality by itself; (3) leadership is a contagious singleness of purpose; (4) it has determination and (5) it exhibits confidence of success. Mr. Currie concluded his talk with some "don'ts" and "do's." He suggested one should not seek popularity and one should not be easy-going. On the other hand, one should be fair and one should remember to encourage people by praising them when praise is due.

A fine ladies' program was developed which consisted of a luncheon and style show and a bus trip to Timberline Lodge on Mount Hood. Unfortunately, the weather was rather inclement (the Conference Arrangements Committee managed to do everything quite well except control the weather) so the ladies were not able to enjoy the splendid scenery during the tour.

There were three interesting mill tours arranged: one was a tour to the Longview Fibre Co. pulp mill at Longview, Wash., and to the lumbering operation of Weyerhaeuser Co. also at Longview; another tour covered Pennsalt Chemicals Corp., Portland, Ore., and the Electric Steel Foundry, also in Portland; and the remaining tour was a trip to Crown-Zellerbach Corp. at Camas, Wash.

Mayor Schrunk of Portland gave a "Welcome to Portland" address at a "Wake-em-up" Breakfast on Tuesday, August 23, and the men attending the conference were treated to a skit "Bad Day at Black Rock" presented by the International Brotherhood of Migratory Peddlers, Waiting Room No. 1.

Sulfite Pulping Session, left to right: P. J. Frost, Crown-Zellerbach Corp., Camas, Wash.; N. Sanyer, U. S. Forest Products Laboratory, Madison, Wis.; C. A. Richardson, Blandin Paper Co., Grand Rapids, Mich.; R. K. Strapp, Sprout-Waldron Co., Pt. Claire, Que.; P. E. Trout, Waldorf Paper Products Co., St. Paul, Minn.

Conference Committee, left to right: A. H. Wickett, local arrangements chairman, Weyerhaeuser Co., Longview, Wash.; D. Wetherhorn, general conference chairman and chairman of the Alkaline Pulping Committee, Continental Can Co., Port Wentworth, Ga.; P. H. West, technical program chairman and vice-chairman of Alkaline Pulping Committee, Thilmany Pulp & Paper Co., Kaukauna, Wis.

SEE HOW THESE PHOTO-MICROGRAPHS PROVE...

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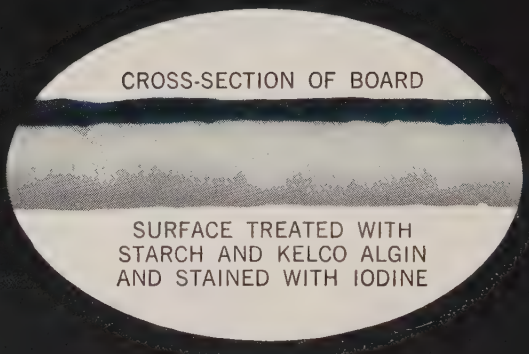

These unretouched photographs show greatly magnified **cross-sections** of two pieces of board: One surface sized with starch alone; the other sized with starch and Kelco algin. See how the starch has saturated through the entire paper on which it alone was used. But what a difference when starch is combined with Kelco algin. This combination remains on the surface, proving beyond question that algin **with** starch controls penetration and strike through. For, with unique Kelco colloids you regulate surface density and penetration, precisely and effectively. This is a positive, reliable fact, providing benefits that are well worth test and evaluation in your own operation.

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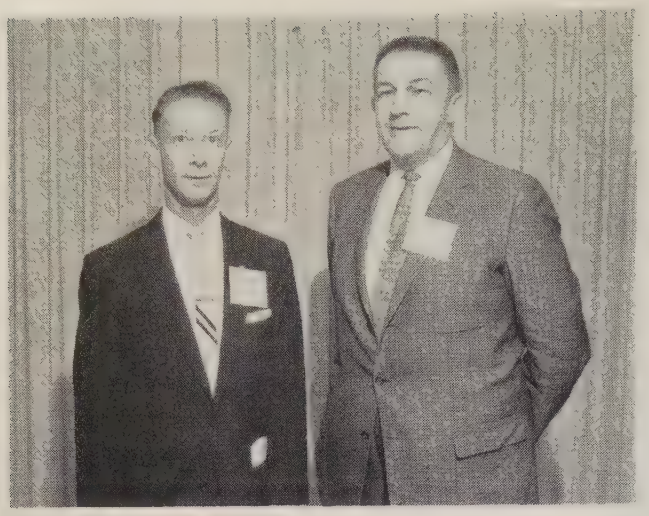


14th Alkaline Pulping Conference chairman and vice-chairman, left to right: D. Wetherhorn, chairman, Continental Can Co., Port Wentworth, Ga.; P. H. West, vice-chairman, Thilmany Pulp & Paper Co., Kaukauna, Wis.

The repertory of the IBMP is quite amazing and each performance is always a most hilarious occasion.

ABSTRACTS

A-1. "An Evaluation of Certain Laboratory Pulping Methods," E. F. Thode, J. R. Peckham and E. J. Daleski, The Institute of Paper Chemistry, Appleton, Wis.

Within the past few years the "traditional" one- or two-cubic foot vertical stationery digester has been supplemented, in many pulping laboratories, by a variety of other pressure vessels. While the newer devices may not be realistic models of commercial-size equipment, they are more efficient in operation than the vertical digester for certain types of experimentation. This is particularly true with "screening experiments" where very small samples of wood are available. Kinetic studies are another field in which efficient special-purpose digesters are needed.

In this paper two such laboratory devices, (1) a "multi-unit digester" comprising seven tumbling autoclaves of approximately one-half liter capacity and (2) an "isothermal digester" comprising a 20-liter stationary pressure vessel of 1000 p.s.i.g. working pressure, are described and compared with conventional equipment.

14th Alkaline Pulping Conference banquet, left to right: A. H. Wickett, Weyerhaeuser Co., Longview, Wash.; P. E. Trout, Waldorf Paper Products Co., St. Paul, Minn.; J. N. McGovern, Parsons & Whittemore, New York, N. Y.; D. Currie, Weyerhaeuser Timber Co., Longview, Wash.

14th Alkaline Pulping Conference banquet, left to right: A. J. Winchester, TAPPI, New York, N. Y.; P. J. Frost, Crown-Zellerbach Corp., Camas, Wash.; E. S. Rice, St. Regis Paper Co., Jacksonville, Fla.; A. H. Wickett, Weyerhaeuser Co., Longview, Wash.

Comparison studies demonstrate that the generalized results obtained with the three devices (in terms of lignin-carbohydrate ratio vs. yield) are essentially similar, chemically speaking. Duplication of chemical and physical results between two different devices is fair to excellent, depending upon how well the temperature and concentration conditions in one may be duplicated in another.

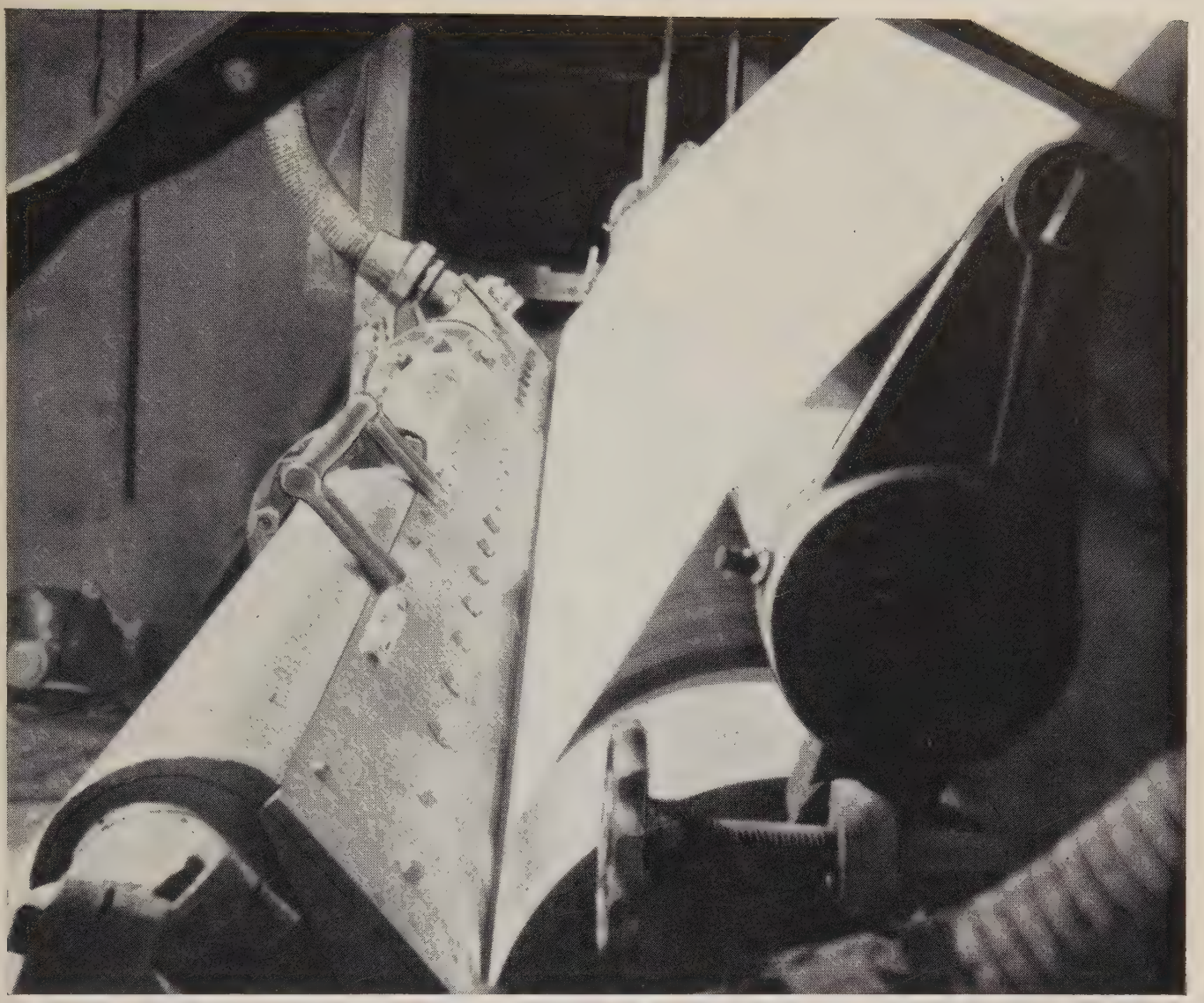
It is recommended that any special-purpose laboratory digester be checked against conventional equipment in terms of generalized response of the chemical processing variables. If such response is consistent with applicable theory, the specialized device may be advantageously employed in experimentation even though its conditions of operation cannot be matched in practice.

A-2. "The Effects of Outside Storage on Slash Pine Chips in the South," C. W. Rothrock, St. Regis Paper Co., Pensacola, Fla., W. R. Smith, Southeastern Forest Experiment Station, Asheville, N. C., and R. M. Lindgren, Forest Products Laboratory, Madison, Wis.

Outside storage of wood chips has been a subject of increasing interest to the paper industry in the South.

Potential advantages to be gained include better wood

14th Alkaline Pulping Conference banquet, left to right: D. Wetherhorn, Continental Can Co., Port Wentworth, Ga.; J. R. Lientz, Union-Bag Camp Paper Corp., Savannah, Ga.; P. T. Dickie, Crown-Zellerbach Corp., West Linn, Ore.; P. H. West, Thilmany Pulp & Paper Co., Kaukauna, Wis.



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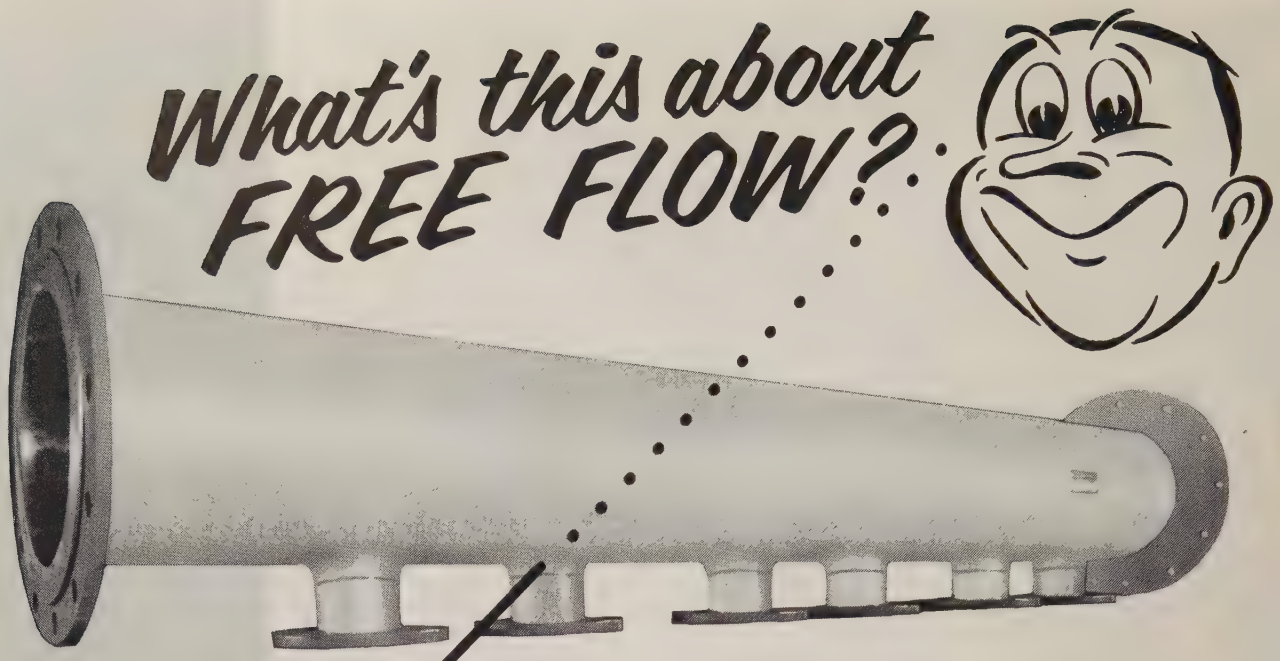

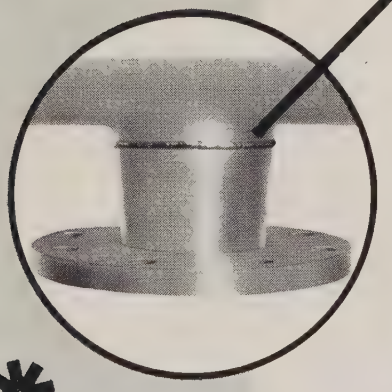

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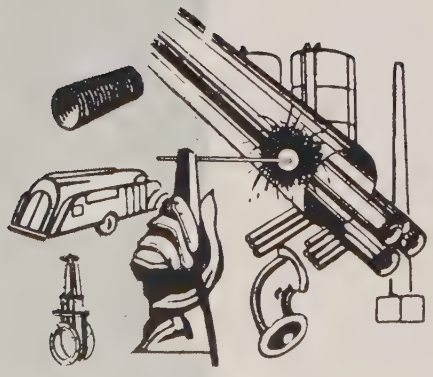

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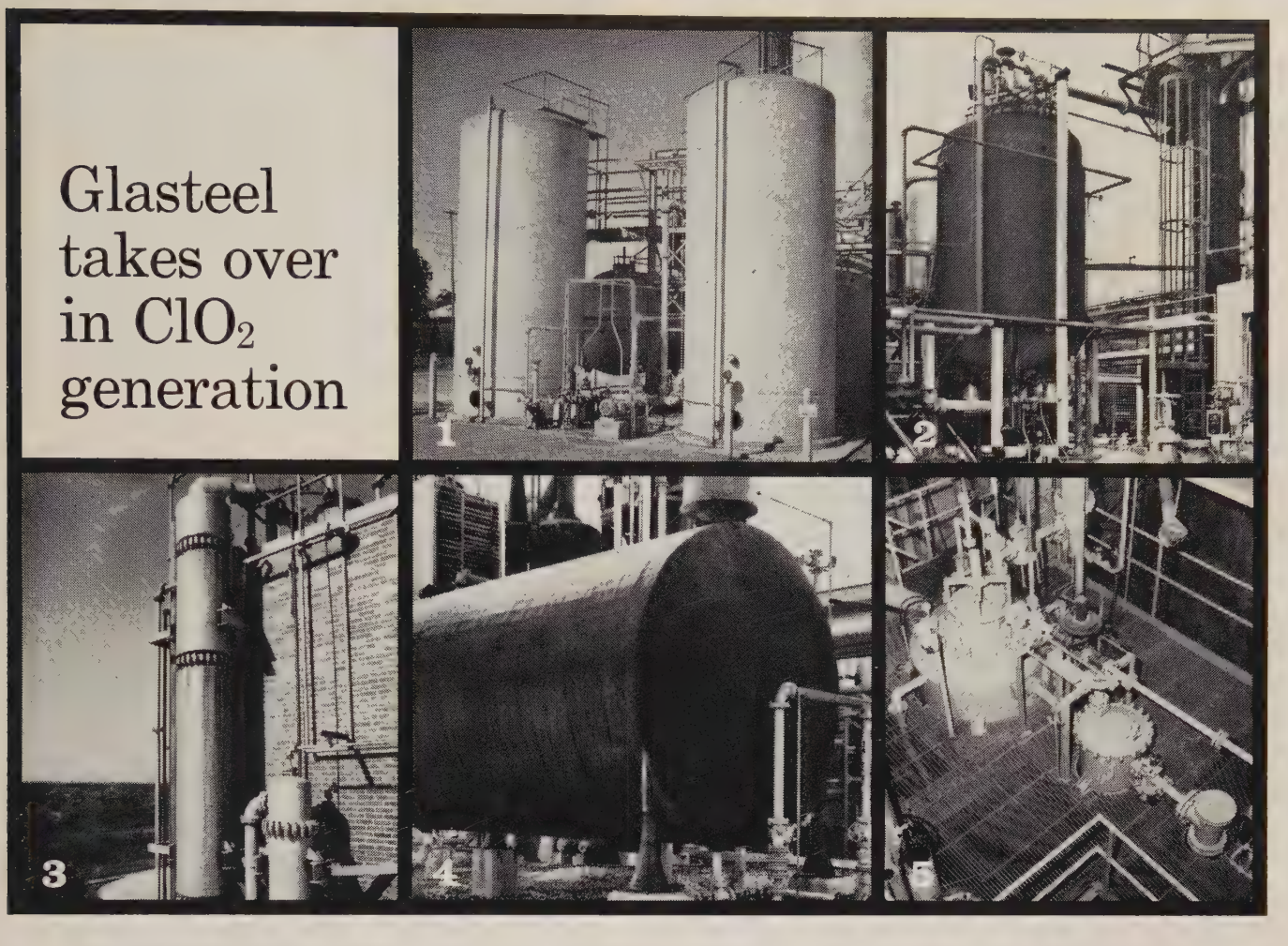

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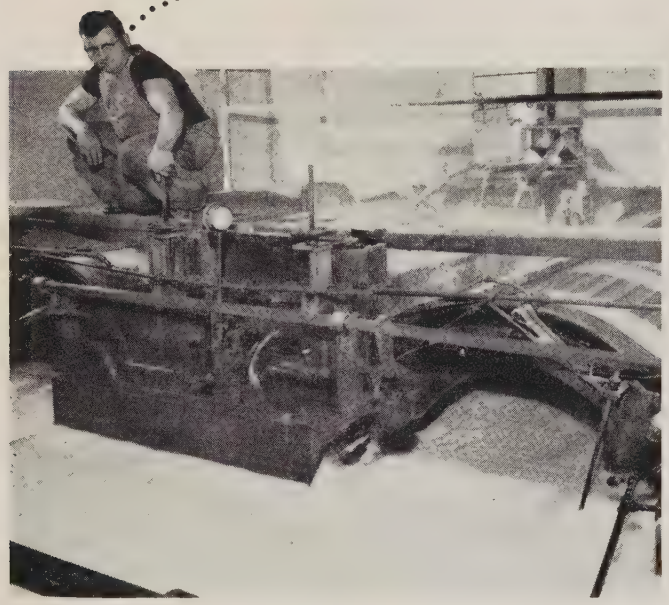

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14th Alkaline Pulping Conference banquet, left to right: P. H. West, Thilmany Pulp & Paper Co., Kaukauna, Wis.; K. M. Shold, Crown-Zellerbach Corp., Port Townsend, Wash.; R. R. Fuller, Gulf States Paper Corp., Tuscaloosa, Ala.; H. O. Teeple, TAPPI, New York, N. Y.

measurement, lower handling costs, stabilized chipping and woodyard operation and solution of storage problems arising from increased use of sawmill residues, mixed species, and establishment of chip mills at the wood source.

A test pile, containing 106 units of slash pine chips, was constructed in June, 1959, at Fargo, Ga. Monthly samples were collected by the Forest Service for moisture and specific gravity determinations and for identification of decay organisms. Samples were also collected by the Development group at Pensacola, Fla., for pulping evaluations.

Repetitive tests were made at each sampling period to insure statistical significance of the results.

Temperatures within the pile increased as much as 60°F. in the first few weeks and remained above ambient levels. A drying cycle in the pile was followed by an increase in moisture to an abnormal level. Dark staining of the chips became evident after two months of storage and was prevalent in about half the chips after five months. Dark stained wood showed a type of fungus attack known as soft-rot. Specific gravity losses amounted to 1–1.5% per month, no greater than observed losses on roundwood.

There was no loss in per cent yield nor change in permanganate number with storage time but the per cent rejects decreased.

14th Alkaline Pulping Conference banquet: A. H. Wickett, Weyerhaeuser Co., Longview, Wash.

14th Alkaline Pulping Conference banquet: D. Wetherhorn, Continental Can Co., Port Wentworth, Ga.

Loss in pulp strength (tear) amounted to about 5% per month, comparable to roundwood.

The results indicate that outside storage of chips in the South is feasible.

A-3. "Experience with the Upflow Continuous Digester," R. H. Rasch, Hammermill Paper Co., Erie, Pa., and L. A. Carlsmith, Improved Machinery, Inc., Nashua, N. H.

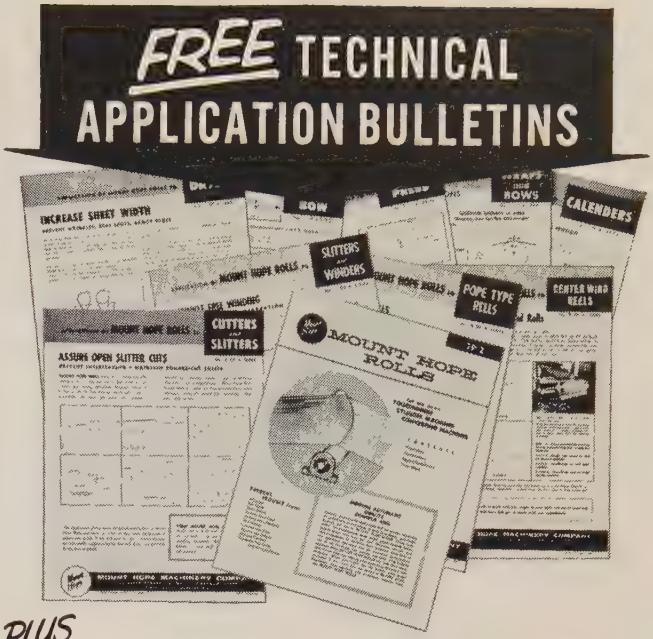
The Impco upflow continuous digester at Hammermill has been in production on NSSC hardwood pulp for about a year. A review is given of the reasons for selection of this type of digester and the characteristics of the pulp made in it. Uniformity of the pulp has been good, considering the wide variations which occur in the proportions of species within the mixed hardwood chip feed. Uniformity within the digester is excellent. Pulp quality is the same within the limits of measurement as batch pulp produced during the same period. The continuous digester takes advantage of the rapid penetration rate of the process, however, investigation of this aspect revealed that chip length must be controlled below $^3/_4$ in. in both batch and continuous digesters. Factors controlling continuous digester capacity are discussed. At increased reaction rates a loss in yield occurs which requires that an economic balance be found between digester capital cost and yield of pulp from wood. The actual retention time within the digester has been measured by substituting spruce chips for the hardwood for a short period and using fiber analysis to determine when these reached the blow cyclone.

A-4. "Diagnosis of Deposits of Pulp Mill Origin," R. T. Mashburn, P. D. Applegate, and H. R. Miller, Hercules Powder Co., Kalamazoo, Mich.

A rational treatment of pulp and paper mill deposits problems consists of diagnosis of the trouble, prescribing a treatment, and removing the cause. This discussion deals with the characterization of deposits, particularly those of kraft pulp mill origin. Attention is focused on the role of deposit analysis in diagnosis.

Likely causes of pulp and paper mill deposits are briefly reviewed along with significant differences in composition which might help to indicate these causes. A systematic scheme for the analysis of deposits is presented. This scheme is intended to identify the individual causes of deposits originating in the pulp mill and of those arising in the paper mill.

Infrared absorption data on the extractables from ten kraft pulps, one NSSC pulp, and one cold soda pulp are presented. These curves are compared with the curve for a



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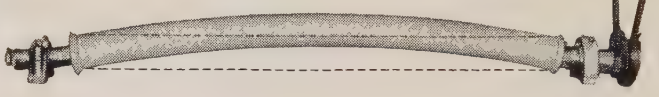

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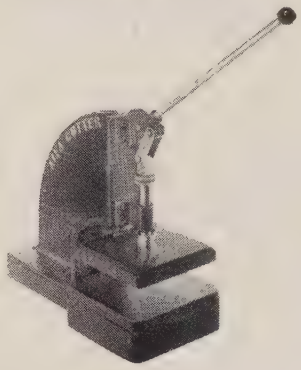

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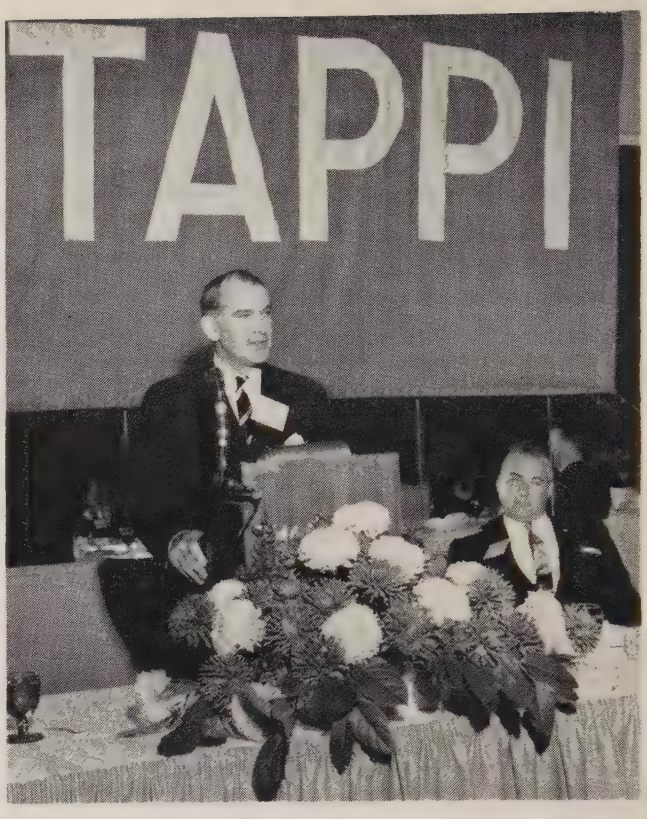

14th Alkaline Pulping Conference banquet: ***D. B. Currie, vice-president, Weyerhaeuser Co., Longview, Wash.***

known mixture comprising resin acids, fatty acids, and neutrals. The value of this tool in identifying major components of deposist is discussed.

Typical case histories employing these methods are cited.

A-5. "Abatement of Air and Water Pollution," V. W. Bacon, Northwest Pulp & Paper Association, Tacoma, Wash.

A-6. "Interactions of Smelt and Water," C. E. Rogers, Babcock and Wilcox Co., New York, N. Y., H. P. Markant and H. Dluehosh, Babcock & Wilcox Co., Alliance, Ohio

An investigation was undertaken to study the physical and chemical reactions involved when a liquid comes into intimate contact with molten smelt. Synthetic smelts of sodium carbonate, sodium sulfate, sodium sulfide, and mixtures of these were produced in a small gas-fired furnace and then brought in contact with water on green liquor. This invariably effected a reaction of explosive proportions whose magnitude depended on the concentration and temperature of the sodium sulfide of the smelt. The chemistry of these explosions involves the generation of hydrogen according to the typical equation for the sodium sulfide water reaction: $Na_2S + 4H_2O \rightarrow 4H_2 + Na_2SO_4$.

A-7. "Experiences with the Production of Chlorine Dioxide," T. C. Payne and B. W. Smith, International Paper Co., Moss Point, Miss.

When it became necessary to increase the production capacity of our Mathieson type chlorine dioxide plant, it was imperative that, in conjunction with the expansion, every effort should be made toward improved chemical efficiencies. To answer both the demands for increased capacity and for improved efficiency, a new 6 tons per day Mathieson type generator was installed as a primary generator with the old 3 tons per day generator as a secondary. This unit arrangement allows the spent liquor at a relatively high sodium chlorate concentration to discharge from the

primary generator to the secondary generator. Here the reaction continues, with the addition of sulfur dioxide, until a relatively low sodium chlorate concentration is reached in the spent liquor effluent. This major change along with other important changes in the absorption tower, water cooling unit, air supply, and storage facilities produced very pleasing results: good plant operation at high production rates, together with an approximate 9% decrease in the sodium chlorate requirement.

A-8. "Operational Experiences with Continuous Calcium Hypochlorite Systems," J. W. Klein, Longview Fibre Co., Longview, Wash., and H. S. Fisher, Pennsalt Chemicals, Tacoma, Wash.

A review and critical analysis of the bleach liquor manufacturing systems at the Longview Fibre Co. from batch through a manual continuous operation to the present automatic system.

In 1954 the first continuous system involving automatic control of lime dilution but manual proportioning of chlorine to lime flows was installed. Chlorination was incomplete with the excess lime used for buffering the hypochlorite bleaching stage. Bleach liquor was unclarified except for screening. This was developed into the present system which incorporates automatic lime dilution, a draft tube type chlorine reactor, oxidation reduction chlorine control and centrifugal cleaning of the finished liquor.

Flow diagrams for each phase are presented together with operating data. Bleach liquor quality in terms of uniformity, clarity and chemical analysis together with chemical losses is discussed. Comparisons are drawn with similar bleach liquor systems operating in the area. A revised design to overcome current operating problems and to provide increased capacity is included.

S-1. "Multistage Sulfite Pulping of Jack Pine, Balsam Fir, and Oak," N. Sanyer, E. L. Keller, and G. H. Chidester, Division of Pulp and Paper, Forest Products Laboratory, Forest Service, U. S. Dept. of Agriculture

Jack pine, balsam fir, and mixed northern oaks were cooked by several multistage sulfite processes in order to compare the processes. Initial cooking took place at a pH of either 3.5 or 6, with liquid sulfur dioxide or soda ash solution injected to complete the pulping. Of the two-stage processes, the best strength resulted when the chips were cooked at about pH 3.5 in the first stage, followed by a neutral sulfite stage. The strength of the pulp approached that of kraft under some conditions. The sulfite pulps, of course, were much brighter than kraft and beat much more rapidly. However, the bisulfite-sulfur dioxide sequence gave somewhat greater yields. Cooking at a pH close to 6 during the initial stage gave 6 to 8 per cent more yield of pulp based on wood because of the increased retention of hemicellulose, but these pulps were weaker than those produced by a bisulfite-neutral sulfite digestion.

In some instances, the shrinkage of pulp on bleaching was greater than anticipated. Pulps of permanganate no. 20 bleached readily in a three-stage treatment with chlorine, caustic soda, and chlorine dioxide, but at permanganate no. 30 it required a five-stage

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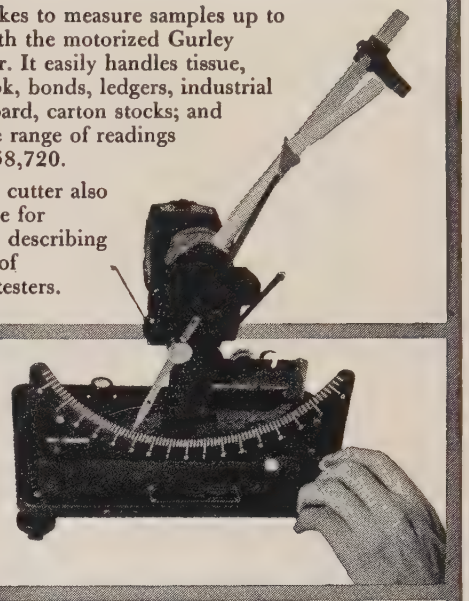

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February

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bleaching sequence including two chlorine dioxide stages. Carbohydrate composition and pitch content of the pulps were compared.

S-2. **"Ultra High Yield Neutral Sulfite Pulp from Balsam Fir,"** C. A. Richardson, Blandin Paper Co., Grand Rapids, Minn.

A continuous installation for making ultra high-yield-neutral sulfite chemimechanical pulp from balsam fir is described.

Small chips are fed continuously to a Messing-Durkee digester equipped with Grenco inlet and discharge valves. The chips reimpregnated for about 15 minutes at 280°F. and 85 p.s.i. with a neutral sulfite liquor composed of a mixture of sodium sulfite and sodium bisulfite. The chips are then treated in a pressafiner and reduced to pulp by two stages of double-disk refining. The pulp is then given a first stage bleach with 0.40/0 sodium hydrosulfite, washed, centricleaned, deckered, and then bleached to 69 brightness with peroxides in a medium density bleach plant. After neutralization the pulp goes through a pump-through refiner to storage, ready for blending ground wood and refined chemical pulps for making coating base stock and printing papers, the pulp is substituted for sulfite pulp to the extent of 20% of the fiber furnish (or about one-half of the chemical pulp) with an increase in bulk and opacity about the same burst and slightly lower tear as compared to the original sheet.

S-3. **"High Yield Sulfite Pulping,"** R. K. Strapp, Sprout-Waldron Co., Ltd., Pte. Claire, Que.

The sulfite process has been long established and has continued to expand in face of competition from other processes. A large tonnage outlet is as reinforcing fiber for newsprint manufacture and the process is adapting itself to competitive pressure in this use through the replacement of news grade by high yield sulfite. Whereas, early high yield sulfite installations did not prosper due to insufficient data, inadequate mechanical devices and operating experience, the latest installation has come smoothly on stream without adverse effect on paper machine operation or newsprint acceptance by the publishers. Further improvement of the process is expected as more operating experience is gained.

As a generality it is stated that savings in chemicals and steam are able to offset the cost of the power used in fiberizing. Wood savings approximate one-half cord per ton of high yield fiber and in some mills will amount in value to more than a half million dollars a year.

This high sulfite yield fiber is seen as a move toward the production of a reinforcing fiber with groundwood-like characteristics and may spur the development of improvements in the groundwood fiber itself. Speculation is also made that bleaching or chemical refining of this fiber may give rise to pulps with enhanced characteristics.

S-4. **"Continuous Semichemical Pulping of Aspen by the Magnefite Process,"** P. E. Trout, Waldorf Paper Products Co., St. Paul, Minn.

The St. Paul mill of Waldorf Paper Products Co. is entirely surrounded by the cities of Minneapolis and St. Paul. Evaluation of chemicals recovery processes for the semichemical pulp mill necessarily required special consideration of the odor level of each process. One process suggested to circumvent the "kraft-type" odors accompanying most sodium-base recovery processes was the Magnefite pulping and recovery process.

Commercial continuous pulping trials were made using a magnesium-base sulfite semichemical process. Both corprugating medium and bleachable grades of semichemical pulp were prepared from aspen wood. Although the desired pulping conditions were not realized completely, the data obtained indicated the following:

1. The Magnefite process will yield a corrugating medium grade of semichemical pulp fully equivalent to that currently produced with the neutral sodium sulfite semichemical process employed by the Waldorf Paper Products Co.

2. The Magnefite process will give a bleachable grade of semichemical pulp having somewhat lower bleached pulp bursting resistance and tearing resistance than the normal bleachable neutral sodium sulfite semichemical process. This is attributed primarily to a greater degree of removal of strength-contributing hemicelluloses by the acid magnesium sulfite process.

3. A neutral magnesium sulfite cook was not practical because of the low solubility of magnesium monosulfite.

4. The extreme corrosivity of the acid magnesium sulfite liquor requires the use of acid-resistant materials of construction throughout the liquor-making and pulping facilities.

TAPPI NOTES

TAPPI Project Appropriations

A SHORT TIME AGO each TAPPI member received a copy of the report entitled "TAPPI Sponsored Research, Its Objectives and Accomplishments." This was prepared to commemorate the 25th anniversary of TAPPI research, to recognize the efforts of those men who contributed vitally to its success, and to evaluate the committee projects that have been sponsored in this period.

Like the "1959 Report to Members" published earlier in the year, this second report was distributed to all members, so that they might be better informed of the Association's continuing efforts to attain its stated objectives. TAPPI can never fully realize those objectives relating to research without the active support of informed members to convert research needs to programs and programs to constructive results.

In addition to reviewing events of the past 25 years the report describes the present operating procedures of the Project Appropriations Committee, as a guide to committees seeking financial support for their projects.

Reference is also made to a 1959 appraisal of the Association's sponsored projects and research programs by a special Research Policy Committee. This committee consisting of J. R. Curtis, chairman, J. H. Heuer, F. S. Klein, and J. L. Parsons was appointed to formulate a research policy which would realistically serve the best interests of TAPPI's individual members and the industry within the organizational and financial structure of the Association.

In preparing its report to the Executive Committee the Research Policy Committee was aided greatly by replies received to an extensive research questionnaire submitted to division chairmen, local section chairmen, and a number of outstanding leaders in association and industry research.

The recommendations of this committee were subsequently approved by the Executive Committee. Since these have direct influence on the sponsorship of present and future projects, it is appropriate to summarize them at this time.

The Association will continue to accept the 1957 "Statement of TAPPI Research Policy" as the primary statement of policy for project appropriations. This statement appears in the chapter on "Regulations Governing Project Appropriations" in the current Yearbook. It places first consideration on committee project activity rather than emphasizing long range research. It grants a wide range of freedom to the Project Appropriations Committee for the allocation of TAPPI funds to any activity or project in the best interests of the membership and the industry. Final authority for the allocation of those funds to be administered by the Project Appropriations Committee is vested in the Executive Committee of TAPPI.

TAPPI's support at this time should be limited to the short-range programs of the committee project type. The object of these programs should be to promote the professional and economic welfare of TAPPI members. Information pertinent to the pulp and paper industry, which is of principal interest to individual TAPPI members, will indirectly benefit companies in most cases. Information of primary interest at the company level, however, may not necessarily benefit the individual. In cases where a distinction is possible, TAPPI will give first consideration in its short-range programs to the interests of the individual member.

Funds to cover the short range, committee project activities will be derived from the TAPPI dues of corporate and sustaining members, as allocated by the Executive Committee.

There should be no substantial increase in the current level of expenditures for project activities. Expenditures for committee work and services of a nonproject nature will be administered through the TAPPI headquarters office from a special fund provided for this purpose by the Executive Committee

The title of the Research Appropriations Committee is to be changed to the more general title of Project Appropriations Committee to cover all project activities. These will be understood to cover both research projects aimed at the development of basic or fundamental information and general projects in the field of applied technology.

There will be no carryover of appropriated project funds past the specified time limits. Project appropriations will be limited to approximately one year. Applications for continuation or renewal of an appropriation will be considered by the TAPPI Project Appropriations Committee on the same basis as a new request.

Division and committee chairmen are advised to exercise close control over their projects covered by TAPPI appropriations. The appointment of divisional "Project Advisory Committees" and the use of specific policies similar to those in the Engineering Division Policy Manual are strongly urged.

At an appropriate time the president should initiate a study to develop an Association policy regarding patents which might result from committee projects and research work.

At an appropriate time the president should appoint a Research Advisory Council to define and develop a program of major long range TAPPI research studies to be supported by the industry rather than the Association. This study should include consideration of the merits of various methods of administering such a program of long-range research, such as a separate research foundation or an expanded Project Appropriations Committee.

PHILIP E. NETHERCUT
Executive Secretary

PERSONAL MENTION

New TAPPI Members

Lester L. Bosch, Partner, Bosch & LaTour, Cincinnati, Ohio, a 1925 graduate of University of Cincinnati.

Robert L. Bowen, Market Analyst, Tennessee Corp., Atlanta, Ga., a 1953 graduate of Georgia Institute of Technology.

Franco Calligaris, Production Manager, Societa delle Cartiere Meridionali, Isola del Liri, Frosinone, Italy, a 1955 graduate of Univerista di Pavia, with a Ph.D. degree.

Basil J. R. Carey, Personal Assistant to Technical Director, Wiggins, Teape & Co., Ltd., London, England.

Woodrow W. Cerf, Project Leader, General Foods Corp., Corn Mill Operations, Kankakee, Ill., a 1940 graduate of University of Louisville.

John P. Churchill, Research Engineer, Champion Paper & Fibre Co., Hamilton, Ohio, a 1956 graduate of New York State College of Forestry.

Gustavo Delfino, Jr., Assistant Manager, Manufacturas De Papel C.A., Caracas, Venezuela, a 1954 graduate of Syracuse University.

Jorge Estrada, Administrator, Celanese Mexicana, S.A., Mexico D.F., Mexico, a 1936 graduate of University of California.

Percy W. R. Gatliff, Chief Machine Control Engineer, English Electric Co., Ltd., Stafford, England, a 1947 graduate of London University.

Jose' J. Gil De Las Casas, Pulp Mill Superintendent, Technica Cubana, S.A., Cardenas, Cuba, a 1953 graduate of Oriente University.

Earl N. Gleaton, Technician, Laboratory Supervisor, Herty Foundation, Savannah, Ga., a 1954 graduate of University of Georgia.

Gary C. Grinnell, Chemist, Nashua Corp., Nashua, N.H., a 1957 graduate of New York State College of Forestry.

Frederick L. Hecklau, Chemist, E. I. du Pont de Nemours & Co., Wilmington, Del., a 1952 graduate of University of Connecticut.

Lillian Hedstrand, Serials Librarian, University of Missouri, Columbia, Mo.

Jean Georges Henry, Assistant Plant Superintendent, Papeteries De Ledar, St. Girons, Ariege, France.

Charles R. Jelm, President, American Alloy Corp., Cleveland, Ohio, a 1942 graduate of Massachusetts Institute of Technology.

Bruce R. Jones, Manager, Research & Development, U. S. Plywood Corp., Redding, Calif., a 1954 graduate of University of Michigan.

Carlos Jorquera H., Superintendent, Cia Manufacturera De Papeles Y Cartones, Santiago, Chile, a 1950 graduate of University of Chile.

George J. Kopp, Sales Engineer, Bauer Bros. Co., Springfield, Ohio, a 1955 graduate of Wittenberg College.

A. Fred Landsberg, Technical Superintendent, Fibreboard Paper Products Corp., Los Angeles, Calif., a 1942 graduate of Technical University Laroverk, Sweden.

Herberi J. Leavitt, Marketing Specialist, General Electric Co., Waterford, N. Y., a 1940 graduate of City College of New York.

Guido Magrin, Production Manager, Societa della Cartiere Meridionali, Isola del Liri, Frosinone, Italy, a 1959 graduate of Technical University, Torino.

Richard A. Martyn, Student, University of Maine, Orono Me.

Morris P. Merchant, Assistant Technical Director, Container Corp. of America, Brewton, Ala., a 1949 graduate of Purdue University with a Ph.D. degree in 1959 from Institute of Paper Chemistry.

Clement R. Moore, Plant Engineer, Moulded Products, Inc., Maple Plain, Minn., a 1954 graduate of University of Minnesota.

M. V. Krishna Murthy, Industrial Relations Officer, West Coast Paper Mills, Ltd., Dandeli, Mysore State, India, a 1938 graduate of University of Mysore.

Ronald E. Nickerson, Plant Engineer, Halifax Power & Pulp Co., Ltd., Sheet Harbor, N.S., a 1949 graduate of Acadia University.

Jean-Paul Pelissier, Chemical Engineer, Procida, Marseille, France, a graduate of Marseille Engineering College with a D.s. degree.

Giuseppe Pelegrini, Chemist, Technical Research, Cartiere Ambrogio Binda, Miland, Italy, a 1912 graduate of University of Bologna and Torino with a Ph.D. degree.

Antonio M. Rivas, Technical Superintendent, Ducilo S.A.I.C., Buenos Aires, Argentina, a 1946 graduate of Buenos Aires University with a Ph.D. degree.

John Robson, Chief Chemist, Adhesives Div., Johnson & Johnson (Gt. Britain), Ltd., Portsmouth, Hampshire England, a 1954 graduate of London University.

Charley J. Rogers, Research Forester, St. Regis Paper Co., Jacksonville, Fla., a 1951 graduate of Michigan State University.

Tore Safholm, Laboratory Manager, AB Casco, Stockholm, Sweden, a 1935 graduate of Royal Technical University of Stockholm.

Albert E. Shaffer, Project Engineer, Consolidated Paper Co.,

Ruth B. Steele, Librarian, Benz Technical Library, Minnesota Mining & Mfg. Co., St. Paul, Minn.

Frederick A. Taska, Sales Engineer, Clark & Vicario Corp., No. Tarrytown, N. Y., a 1949 graduate of Fairleigh Dickinson University.

Thomas C. Theiler, Divisional Staff Engineer, Owens Illinois Glass Co., Toledo, Ohio, a 1953 graduate of Purdue University.

Vinayakrai J. Vaishnav, Technical Officer, Imperial Chemical Industries (India) Private, Ltd., Bombay, India, a 1936 graduate of Baroda College.

Wolfgang O. Wilfert, Superintendent Coating Dept., Riegel Paper Corp., Milford, N. J., a 1942 graduate of Air Force Academy.

Raymond O. Witte, Superintendent, Southern Engineering Dept., The Employers Group of Insurance Companies, Atlanta, Ga.

Franklin D. Wittmer, Chemical Process Research Engineer, A. H. Lundberg & Associates, Inc., Mercer Island, Wash., a 1955 graduate of Michigan College of Mining and Technology.

Robert L. Woodall, Student, Western Michigan University, Kalamazoo, Mich.

Takeo Yada, Managing Director, Showa Jushi Kogyo, Ltd., Osaka-Pref., Japan, a 1933 graduate of Kanazawa University of Chemistry.

Tetsuzo Yamada, Director, Dainichi Electric Wire & Cable Co., Ltd., Amagasaki, Japan, a 1932 graduate of Kyoto University.

Isao Yamamoto, Superintendent of Production, Sanzen

Seishi Co., Ltd., Kanazawa, Japan, a 1948 graduate of Shinkyo Technical College.

John R. Young, Research Manager, Paper Group, Metal Box Co., Ltd., London, England, a 1944 graduate of London University with a Ph.D. degree in 1951.

Appointments

Adrien W. Allard, formerly of the Nashua Corp., is now Chemist for Bloomingdale Rubber Co., Aberdeen, Md.

Carrol Allen is now Vice-President in charge of fiber products for Southern Pine Lumber Co., Diboll, Tex.

Richard G. Allen, formerly of Ohio Boxboard Co., is now Chemist in the central laboratory of Packaging Corp. of America, Quincy, Ill.

Nils O. Arve, formerly of Skogsagarnas Industri A/B., is now Works Manager of Morrums Bruk, Morrum, Sweden.

T. C. Bannister, Jr., is Vice-President and General Manager of Bowaters Carolina Corp., Catawba, S. C.

John S. Barton of Crown Zellerbach Corp. has been tranferred from San Leandro, Calif., to the Central Research Division, Camas, Wash., as Director of Research.

Edward W. Beardsley of the Corn Products Sales Co. has been transferred from San Francisco, Calif., to Atlanta, Ga., as Southern Regional Vice-President.

Carlos Benkos, formerly of Cia. Santista de Papel is now Director of Industrias de Papel Rio Verde S/A, Sao Paulo, Brazil.

Murray H. Bennett is now President of Chemical Linings Division of Atlas Mineral Products Co., Mertztown, Pa.

Carroll H. Bessey of Nicolet Industries is now an Industrial Engineer with Nicolet Industries, Florham Park, N. J.

Donald Blackburn of Martin Paper Products, Ltd., has transferred his office from St. Boniface, Man. to Vancouver, B. C.

John C. Braznel of Champion Paper and Fibre Co. has been transferred from Hamilton, Ohio, to St. Louis, Mo., as Manager.

John S. Brush, formerly of The Cuban American Sugar Mills Co., is now assistant Production Manager of Calcasieu Paper Co., Elizabeth, La.

Emmet W. Bryan of the U. S. Corps. of Engineers has been transferred from Savannah, Ga., to Abilene, Tex., as a Mechanical Engineer.

George Burkhard, formerly of The Quebec North Shore Paper Co., is now a Research Engineer for the Dominion Engineering Co., Ltd., Lachine, P. Q.

Philip D. Caine of Becco Sales Corp. has been transferred from Boston, Mass., to New York, N. Y., as Sales Promotion Manager.

Charles J. Campbell of Kimberly-Clark Corp. has been transferred from Neenah, Wis., to West Carrollton, Ohio, as Plant Engineer for the Moraine Paper Division.

Thomas J. Carlin, formerly of the Eastern Corp., is now Kraft Mill Superintendent of Price Brothers Co., Ltd., Keno-gami, P. Q.

R. J. Casey of J. M. Huber Corp. has been transferred from New York, N. Y., to Appleton, Wis.

Marshall J. Charlton of The Forest Products Division, Olin Mathieson Chemical Corp., has been transferred from Joliet, Ill., to West Monroe, La.

Bernard D. Cox, formerly of Gulf States Paper Corp., is now Technical Superintendent of Calcasieu Paper Co., Elizabeth, La.

J. Kenneth Craver of the research and engineering division of Monsanto Chemical Co. has been transferred from Dayton, Ohio, to St Louis, Mo.

J. D. Dailey, formerly of Riegel Carolina Corp., is now Senior Vice-President of Allied Paper Corp., Kalamazoo.

Roy L. Davis, formerly of Cia Industrial de San Cristobal S/A., is now Manager of the West Coast Division of Scott Paper Co., Everett, Wash.

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William M. Delaware, formerly a Student at the University of Maine, is a Chemical Engineer with the International Paper Co., Glens Falls, N. Y.

Richard C. Dempsey, formerly with A. E. Staley Mfg. Co. is now a Technical Sales Representative for Lockport Felt Co., Newfane, N. Y.

Theodore E. Durst, formerly a Student at the University of Maine, is now a Development Engineer for Bird Machine Co., So. Walpole, Mass.

Tage Elers, formerly a Graduate Student at the University of Washington, is now a Graduate Student at the School of Forestry, North Carolina State College, Raleigh, N. C.

Bengt A. G. Ericsson, formerly of the West Virginia Pulp & Paper Co., is now a Mechanical Engineer for A/B Nykvarns Bruk, Nykvarns, Sweden.

Loren V. Forman of the Scott Paper Co. has been transferred from Everett, Wash., to Chester, Pa., as the Executive Assistant to the Vice-President in charge of production, engineering, and research.

J. Hori Freiday is now Assistant Manager for paper mill equipment, Combustion Engineering, Inc., Windsor, Conn.

F. A. Futch, formerly of Venezolana de Pulpa y Papel is now Assistant Superintendent of the paper mill department of Union Bag-Camp Paper Corp., Savannah, Ga.

James J. Harrison is now Vice-President in charge of manufacture for New Haven Board & Carton Co., New Haven, Conn.

John C. Harper, formerly of Rice Barton Corp., is now Sales Engineer for Armstrong Cork Co., Lancaster, Pa.

William M. Hearon is now Vice-President for research and development of Crown Zellerbach Corp., San Francisco,

Jay G. Herr, formerly of The Brown Co., is now a Technical Service Engineer for the St. Regis Paper Co., Sartell, Minn.

Edward R. Hinson ,formerly a Student at the North Carolina State College, is now a Junior Technologist for Champion Paper & Fibre Co., Houston, Tex.

Ian C. Innes, formerly of Bowaters Research & Development, Inc., is now an engineer for Tasman Pulp & Paper Co., Kaweran, New Zealand.

R. S. Jobin, formerly of G. Davidson & Sons, Ltd., is now with Whakatane Board Mills, Ltd., Whakatane, Bay of Plenty, New Zealand.

Arvo Kantola, formerly of Marathon Corp., is now an Engineer with Rauma-Repola Oy, Rauma, Finland.

John L. Keyes, formerly of Powell River Co., Ltd., is now Research Director for MacMillan, Bloedel & Powell River Co., Ltd., Vancouver, B. C.

John B. Koehler, formerly of John B. Koehler & Associates, is now with Koehler Coating Machinery Corp., Greentown, Ohio.

W. P. Lawrence is now Director of Research and Develop ment for Champion Paper & Fibre Co., Hamilton, Ohio.

John L. Lawes of Hercules Powder Co. has been transferred from Los Angeles, Calif., to Chicago, Ill., as a Technical Salesman.

Milton A. Lefevre, formerly of Marathon Corp., is now Technical Director of Fletcher Paper Co., Alpena, Mich.

Benjamin D. Levine, formerly of the Orchard Paper Co., is a Research Project Leader for the Carton & Container Division, General Foods Corp., Battle Creek, Mich.

Malcolm B. Lowe, formerly of the Lowe Paper Co., is now Product Manager of the Riegel Paper Corp., Acme, N. C.

Everett A. Mahannah, formerly of Ebasco Services, Inc., is now a Pulp & Paper Engineer for Roderick O'Donoghue & Co., New York, N. Y.

Sergio Martinez, formerly of Papelera Pulpa Cuba S. A., is now a Project Engineer for Parsons & Whittemore, New York, N. Y.

William A. Moggio, formerly of Rhinelander Paper Co. is now a Research Chemist for Armstrong Cork Co., Lancaster, Pa.

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George E. Motz, formerly of Nopco Chemical Co., is now a Salesman for Arnold,Hoffman & Co., Atlanta, Ga.

Edward K. Nissen, formerly of the Solvay Process Division, is now Head of the Industrial Starch Technical Service, Union Starch & Refining Co., Granite City, Ill.

Harry N. Packard, formerly Student at the University of Maine, is now a Junior Project Engineer for Great Northern Paper Co., Millinocket, Me.

Lester L. R. Phillips of the Hurlbut Paper Co. has been transferred from South Lee, Mass., to Los Angeles, Calif., as Assistant Sales Manager.

Charles E. Price, formerly of Gardner-National Co., is now a Research Project Leader for the The Carton & Container Division, General Foods Corp., Battle Creek, Mich.

Charles J. Priest, formerly with G. A. Venezolana de Pulpa y Papel, is now Coating Supervisor for Continental Can Co., Inc., Augusta, Ga.

Lester J. Randall, formerly of Walter Kidde Engineers-Southwest, Inc., is now an Application Engineer for the General Electric Co., Plainville, Conn.

Samual I. Reames, formerly of Hudson Pulp & Paper Co., is now a Mechanical Engineer with Bowaters Engineering Development Corp., Calhoun, Tenn.

Malcolm J. Rundell, formerly a Student at the University of Maine, is now a Chemical Engineer with the Northwest Paper Co., Cloquet, Minn.

Roy D. Rivers, formerly with Champion Celulose S. A., is now a project manager with the pulp and paper group of M. W. Kellogg Co., New York, N. Y.

George W. Sargent, Jr., formerly of Mercury Engineering Corp. is now a Staff Engineer with Beloit Iron Works.

Charles Schenker is now Pulp Mill Superintendent of Buckeye Cellulose Corp., Foley, Fla.

Raymond W. Snyder, formerly of West Virginia Pulp & Paper Co., is now Quality Control Supervisor of Hamilton Paper Co., Miquon, Pa.

Chester U. Stevens, formerly of Eaton-Dikeman Co., is now a Research Chemist with the Peter J. Schweitzer Division, Kimberly-Clark Co., Lee, Mass.

William A. Strickler, formerly of Pusey & Jones Corp., is now a Salesman for the Beloit Iron Works, Beloit, Wis.

F. A. Strovink is now Eastern Regional Sales Manager for American Cyanamid Corp., New York, N. Y.

Julian E. Tobey, formerly of Appalachian Coals, Inc., is now a Consulting Engineer in the Carew Tonwer, Cincinnati, Ohio.

H. Dale Turner is now Research Manager of Masonite Corp., St. Charles, Ill.

Kalevi Vesterinen, formerly of Enso Gutzeit Oy, is now Technical Manager of the newsprint mill of Jujo Paper Mfg. Co., Ltd., Kushiro City, Hokkaido, Japan.

Jack F. Wade, formerly of Kimberly-Clark Pulp & Paper Co., Ltd., is now Technical Director of Irving Pulp & Paper, Ltd., Lancaster, New Brunswick.

David Wetherhorn is now Technical Director of Southern Paperboard Division, Continental Can Co., Port Wentworth.

James F. Whalen of Becco Chemical Division has been transferred from Chichago, Ill., to Charlotte, N. C.

Robert C. Whitney, formerly of the Brown Co., is now a Project Engineer for Oxford Paper Co., Rumford, Me.

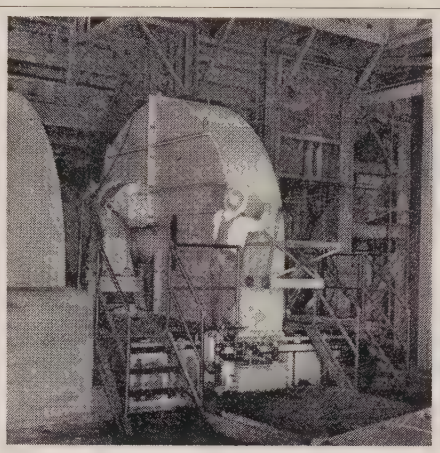
Albert L. Wiley, formerly of Riegel Paper Corp., is now Technical Manager of Scott Paper Co., Mobile, Ala.

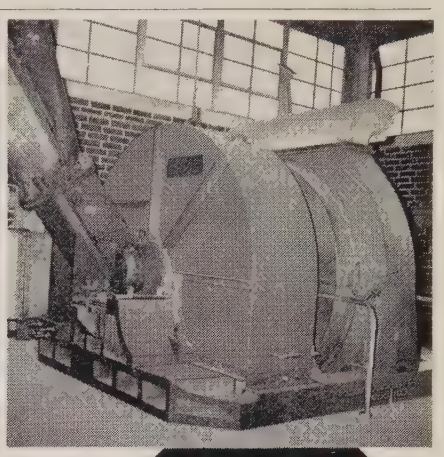
Richard E. Williams, formerly student at New York State College of Forestry, is now a Process Engineer for KVP Sutherland Paper Co., Kalamazoo, Mich.

Murray Wolf, formerly with Times Facsimile Corp., is now Technical Director for Tri-Wall Containers, Inc., New York, N. Y.

William A. Young, Jr., of Hercules Powder Co. has been transferred from Atlanta, Ga., to Wilmington, Del., as a Technical Service Engineer.

Albert P. Yundt is now Assistant to the President of Bird & Son, East Walpole, Mass.





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MURCO Round Log Pulpwood Chippers are designed to include the most desirable features for today's production requirements . . . the result of long and careful study of the various features that contribute to outstanding chipper performance in producing more and better chips at less cost . . . less sawdust, fewer slivers, freedom from repairs yet at the same time having production records of 100 cords or more per hour. The MURCO heavy design construction reduces vibration.

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INDUSTRY NOTES

North American

Statistics

*Paper and Paperboard Production**

Production of paper and paperboard in the first six months of 1960 totaled an estimated 17.5 million tons, up 3.4% from the corresponding year-ago total. First half 1960 production consisted of 7.9 million tons of paper, 8.0 million tons of paperboard, and 1.6 million tons of construction paper and board. Paper and paperboard production were well ahead of year-ago totals, but output of construction paper and paperboard, under the impact of declining residential construction, averaged out below the year-earlier highs. On a seasonally adjusted basis, the trend of paper output during the first half of 1960 was steadily upward, with the rates for the second quarter making new highs. Paperboard production adjusted for seasonal factors fluctuated about a flat trend that was a little below the highs established in the second and third quarters of 1959. The rate of paper and paperboard production was sharply curtailed during the first two weeks of July as a result of usual vacation shutdowns. Normal seasonal factors indicate a 10 to 15% drop in production from the May–June level for the month of July, data for the first two weeks of July suggest that this year the decline will be somewhat larger.

*Pulpwood**

Total receipts of pulpwood for January–May, 1960, were 16,796,000 cords, an increase of 1,864,000 cords over receipts for the comparable period last year. Domestic receipts were 1,789,000 cords above the year earlier total, and imports increased 75,000 cords. All of the regions showe' increases ranging from 8% in South Central and Northeast to 18% in the West region.

Pulpwood consumption for January–May, 1960, was 16,964,000 cords, an increase of 1,005,000 cords over the same period of 1959. All of the regions showed increases ranging from 4% in Northeast and South Central to 13% in the North Central region.

Inventories of pulpwood at the end of May were 4,957,000 cords, a decrease of 328,000 cords below April, 1960, and a 122,000 cords increase above 1959. Two regions showed decreases: North Central decreased 1% and Northeast decreased 17%. Three regions showed increases ranging from 6% in the West to 19% in the South Central.

*Wastepaper and Other Fibrous Materials**

May wastepaper receipts were 754,000 tons, for the first five months of this year they totaled 3,749,000 tons, a decrease of 129,000 tons below receipts for last year.

Wastepaper consumption for May was 770,000 tons, the January–May total was 3,796,000 tons, a decrease of 28,000 tons below consumption for the same period last year.

Inventories at the end of May were 540,000 tons, a decrease of 17,000 tons below April and an increase of 51,000 tons above inventories at the end of May, 1959.

Consumption of total fibrous materials for May was 850,000 tons, the January–May total was 4,213,000 tons, a decrease of 55,000 tons below consumption for the comparable period last year.

Inventories of total fibrous materials at the end of May were 940,000 tons, 42,000 tons below April and 42,000 tons above inventories at the end of May, 1959.

*Wood Pulp**

Production of wood pulp for January–May, 1960, was 10,584,000 tons, an increase of 591,000 tons above the comparable 1959 total.

Wood pulp consumption in the manufacture of paper and board for January–May, 1960, was 10,879,000 tons, an increase of 545,000 tons above consumption for the same period last year.

Imports of wood pulp for January–May, 1960, were 972,256 tons, a decrease of 3% below imports for last year. Imports from Canada for January–May, 1960, were 827,945 tons, an increase of 2% above the comparable period of 1959. Overseas imports for January–May, 1960, were 136,537 tons as compared to 185,181 tons for the same period last year.

Exports of wood pulp for January–May, 1960, were 370,521 tons, as against 233,876 tons exported during the same period last year.

Inventories of wood pulp at paper and board mills at the end of May were 526,279 tons, an increase of 6,000 tons above inventories at the end of April and 29,000 tons below inventories at the end of April and 29,000 tons below inventories at the end of May, 1959.

Census Releases Final 1959 Paper and Board Production Data

Paper and board production for 1959, as issued by the Bureau of the Census on August 18, showed that 34,007,068 tons were produced—15,024,916 tons of paper, 15,456,492 of paperboard, 149,658 of wet machine board, and 3,376,002 tons of construction paper and board. Wisconsin was the largest producer of paper and board, 2,181,752 tons, with Florida second, 1,955,561 tons and New York third, 1,892,553. In all there were 16 states producing more than a million tons of paper and board.

Revised data for 1958 showed a total of 30,814,366 tons—13,488,335 tons of paper, 14,149,930 of paperboard, 121,103 of wet-machine board, and 3,054,998 tons of construction paper and board (Census Report "Pulp, Paper and Board, 1959"—Series M26A-09).

Paper and Papermaking Materials Rank Fourth in Value of 1959 U. S. Imports

Paper and papermaking materials imports in 1959 totaled $1,089,400,000 in 1959, ranking fourth of 30 leading commodity groups. Of the total, newsprint was valued at $665.7 million, paper base stocks $346.1 million and paper manufactures, other than newsprint, $77.6 million. Canada was the largest supplier, imports from that country totaling $959.2 million (U. S. Department of Commerce, World Trade Report, "Leading Commodities in U. S. Foreign Trade, 1959"–June, 1960).

Overseas

Argentina

Paper Firm Seeks Foreign Financial Assistance

Papelera Royal, S.A. (Corodoba 836, Rosario, Argentina) is interested in obtaining financial cooperation in the es-

* American Paper and Pulp Assoc., *Monthly Statistical Summary* 38, No 8 (August, 1960).

tablishment of a pulp and paper mill. The desired cooperation "may take the form of a direct loan, the supply of machinery and equipment or participation in the enterprise by a paper company supplying the needed machinery and equipment."

"The company is desirous of obtaining the equivalent of 15 million pesos (around U. S. $190,000) of foreign capital in order to complete its initial plant, construction on which has now begun."

Pulp and Paper Manufacturers and Converters

Bay West Paper Co.

"Jack-of-All-Trades" Adds Up

In this age of specialization, the old time jack-of-all-trades has been down-graded by the insistence that he is "master of none."

The fact remains, however, that there are many situations in which versatility is essential if the individual is to do his job well. By the same token, desk-top office machines often have to perform in a variety of functions, and the attitude which insists on one machine for one particular job must run into trouble in such circumstances.

Ironically enough, we start with the fact that Bay West Paper Co. is a specialist—a paper converter. We make 12 different items from paper stock supplier by our parent organization Mosinee Paper Co.

Bay West was founded in 1922, and became a Mosinee subsidiary in 1928. Just about six years ago we moved into a new plant with 50,000 sq. ft. of space, yet the volume of business has been such that in that time we have had to double plant capacity.

Any company generates figurework, and ours is no exception. There are not such vast quantities, however, to warrant special treatment for every area in which paperwork might be involved.

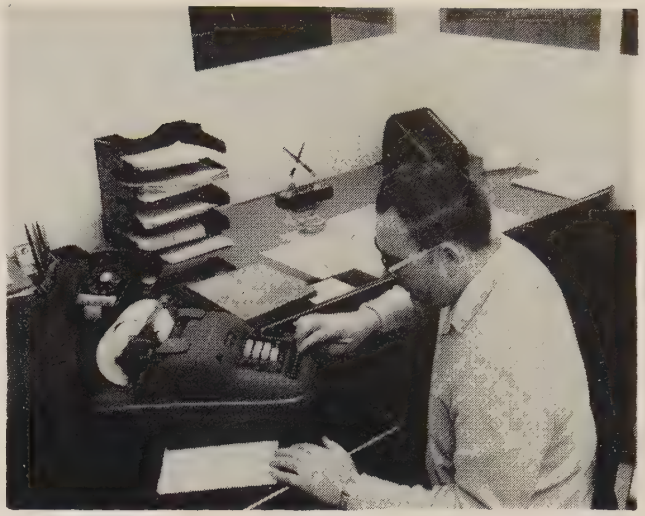
It's at this point that management often makes an understandable error. It is sometimes assumed that since the figurework to be done is not in huge quantities, any machine can do it. This simply is not so. Or, if most of the calculating is done in one particular area, specialized machines are provided which then have limited utility because they cannot readily be used for other jobs.

Specifically, we have a continuing need for adding machines. We also have a continuing need for a calculator. Because of the variety of work to be done, we settled on a "jack-of-all-trades"—the Remington Rand 99 Printing Calculator.

In the production and shipping department, figures must be worked up every day to determine inventory levels, which in turn affect production schedules. Much of this work is simple adding and subtracting, though even here situations sometimes arise where percentages must be determined. As the Printing Calculator automatically multiplies and divides, adds, and subtracts it is an effective multipurpose unit.

Shipping moves goods out to our own railroad dock for shipment to our wholesaler customers; 90% to of our shipments move by rail. These movements, of course, must be covered by bills of lading, which pose a distinctly different figurework routine.

We work on an average weight arrangement. The different items we ship must be totaled on the basis of known weights—Zip towels, for example weigh 28 lb. per box, while Prep towels weigh 30½ lb. per box—and then a total found for each shipment. This is basically a problem in multiplication, quickly handled on the 99 through its automatic multiplication feature.



The Remington Rand 99 Printing Calculator at Bay West Paper Co., Green Bay, Wis. is used as an economical and efficient "jack-of-all-trades." It serves as both calculator and adding machine

An added measure of speed, which is a consideration in this work, is achieved because of the Simpla-tape record printed by the machine. This provides a clear record of every step in the computation. All the operator has to do is to check visually the tape to make sure he entered the correct figures, for then the answer must be correct. There is no need to run the problem through twice.

Speed is essential for various reasons, among them the fact that the Traffic Department uses the shipment figures in order to bring in the necessary freight cars. If five cars are ordered when only four are needed, the extra car represents a dead loss. Similarly, if four are ordered when actually five can be used, some shipments will be held up.

The necessary figurework must, therefore, be completed in time for Traffic to take intelligent action. Time lost can be reflected in unnecessarily high loading costs.

Finally, the same machine is used to prepare our payroll. This is complicated by a bonus system, based on extra production. When regular pay has been determined this bonus is calculated and added to it.

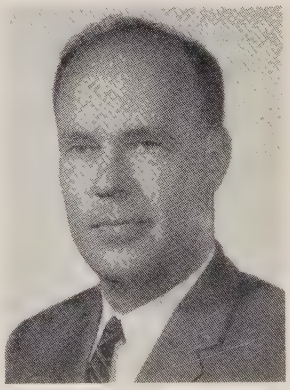
In this case the Simpla-tape becomes a part of the record. It is attached to the man's card and becomes part of his file, instantly available to determine how his total pay was arrived at if the need to check should arise.

Our mechanical "jack-of-all-trades" handles each job as a matter of course, performing far more economically and efficiently than could more specialized units. It was chosen, after careful study, because of its ability to do a wide variety of jobs plus the invaluable printed record of calculations, and has functioned as an effective "workhorse" since we first put it to use.

Bowaters Carolina Corp.

Appointment

T. C. Bannister, Jr., has been appointed a vice-president of Bowaters Carolina Corp., Catawba, S. C. Mr. Bannister, who will continue as general manager of Bowaters Carolina, has responsibility for day-to-day control of all existing and planned operating units at Catawba.



T. C. Bannister, Jr.

Brown Co.

Cordwell Named Sulfite Production Manager

C. A. (Buster) Cordwell has been promoted to the position of sulfite production manager. He has been closely associated with sulfite pulp production since joining the company in 1926 and served in various supervisory positions in the Alpha Plant, sulfite bleachery, and sulfite mill during the past 34 years. He was made superintendent of the mill in 1950.

Container Laboratories, Inc.

Conlab Standards Group

A group of laboratories scattered throughout the United States and Europe have been brought together under a program aimed at more positive assurance that test work is accurately and reliably reproducible within any of the member laboratories. The laboratories are doing test work on paper and paperboard products and on packages and packaging materials of all kinds.

According to James A. Sargeant, president of Chicago-based Container Laboratories, Inc., an independent design, testing, and engineering laboratory which is coordinating the program, as many as 100 laboratories of package and package materials manufacturing companies are expected to participate in the program, to be known as the Conlab Standards Group. The program is already under way with some 40 laboratories actively participating.

Tests will be selected on the basis of ideas received from participating CSG members, and will be run in each member's laboratory. Every month each member laboratory will receive a sealed, moisture-vapor proof package of materials that have been preconditioned to assure uniform equilibrium of moisture content. Each laboratory is assured of receiving materials as physically identical as possible to those received by other participating laboratories. Materials to be tested will have been scientifically random-sampled and the tests will normally be changed each month. They will include flat crush, moisture content, bursting, puncture, moisture vapor transmission, tensile, ply separation, ring stiffness, caliper, basis weight, tear, porosity, smoothness, and compression.

The packet will contain instructions citing applicable testing standards and each participating laboratory will be given a code number which will change each month and which will identify the laboratory in the monthly summary reports. Code numbers of participants will be confidential.

Summary reports will indicate:

1. If all laboratories report results in acceptable correlation with the others, it will mean that in this particular test results obtained in an individual laboratory are truly reproducible with results obtained in other laboratories.

2. If results from an individual laboratory do not correlate with those of other laboratories, every assistance will be given by Container Laboratories in identifying the cause(s) of failure to correlate.

3. If results indicate very poor or completely unacceptable degrees of correlation between the majority of individual laboratories, steps will be taken to find where the fault lies.

There may be too many permissible variables in the procedure standards, too many variables produced by types of equipment used, and other factors that could affect reproducibility of test results.

According to Mr. Sargeant, data obtained from the test work will be used in a number of ways:

1. Establish up-to-the-minute reliable correlation of test work between participating laboratories.

2. Identify variations in laboratory practices which could contribute to poor correlation between participating laboratories.

3. Development of possible useful correlation between different kinds of tests and test devices.

4. Permit member laboratories to objectively test their performance against a group average.

5. Provide an informed basis for recommendations to TAPPI, ASTM trade associations, government and other standards committees concerned with specific test methods, equipment, techniques, and materials.

In addition to the monthly summaries, an annual compilation will be made of all work done. These results will be analyzed and reported to each participating member in an annual publication.

At the end of the first year, and at six-month intervals thereafter, all participating laboratories will be graded on consistency and reliability of correlation and reproducibility of test results for that period. The grading will be confidential, but laboratories that achieve a high standard will be given certificates of excellence.

An annual seminar will be held for laboratory supervisors and other interested in this kind of activity. Member laboratories will receive periodic mailings of loose leaf forms covering practical and proved laboratory operating procedures evolved over the past 37 years by Container Laboratories. These will cover such topics as:

1. Maintenance and operation of laboratory equipment.
2. Step-by-step operation guides.
3. Preparation of specimens and handling them through the testing operations.
4. Pretesting conditioning of specimens.
5. Recording and interpreting data.
6. Presentation of test results.
7. Calibration of laboratory equipment.

Appointment

Frederick C. Meyer has been appointed general manager of Container Corp.'s Boyle Avenue folding carton plant, Los Angeles, Calif.,

William B. Whiting has been appointed a division general manager responsible for Container Corp.'s Philadelphia and Chattanooga paperboard mills. William Erskine will succeed Whiting as general manager of the Philadelphia mill.

The Crossett Co.

Union-Bag Will Not Purchase Crossett

Union Bag-Camp Paper Corp. will not exercise its option to buy The Crossett Co., according to an announcement made recently by P. F. Watzek, president of Crossett.

According to Mr. Watzek, a majority of the present stockholders had decided to withdraw their stock from sale and to continue to operate the company as in the past. Crossett stockholders were also informed that although a thorough investigation of The Crossett Co.'s business and properties by Union Bag-Camp had established the fact that all assets were as represented, the New York firm had not been able to arrange financing on terms satisfactory to it. According to Union Bag-Camp this was due partially to antitrust proceedings started earlier in the summer against the large paper firm by the Federal Trade Commission. While Union Bag-Camp does not feel that the proceedings are correct, they did nevertheless add further uncertainty to the financing.

News of the possible sale of The Crossett Co. to Union Bag-Camp Paper Corp. was first made public last May when it was announced that agreement had been reached whereby the New York firm would "investigate the possibilities of acquiring The Crossett Co. through purchase of its stock at $68.50 per share." Total purchase price would have been $156 million.

Georgia-Pacific

Toledo Kraft Mill Completed

Georgia-Pacific Paper Co., a subsidiary of Georgia-Pacific Corp., formally opened the newly completed second half of its $25-million kraft paper mill at Toledo on August 1.

View of Georgia-Pacific's expanded Toledo, Ore., paper mill shows, left to right: rail spur and warehouse, paper machine room, maintenance building and laboratories, pulp mill, power plant, and chemical reclamation facilities. In foreground is administration

Secretary of State Howell Appling scissored a brown kraft paper ribbon at the plant site, before an audience of 100 state and county officials and paper industry representatives, during brief ceremonies at 10:30 a.m.

The Toledo construction is part of a $60-million expansion program begun in 1954 by the corporation, which is headquartered in Portland, to expand facilities in its 27 plants from coast to coast. Toledo will sell the major portion of its paper and containerboard output in the west.

To ensure a year-round water supply, G-P erected a 70-ft. dam on Olallie creek, impounding water for dry periods and creating a 100-acre fresh water lake, which serves as a recreational and fishing area. Toledo's two paper machines now produce 600 tons daily, from 30 lb. paper to 69 lb. containerboard. The new machine trims to 160 in. and produces kraft paper for wrapping, shopping bags, multiwall bags and numerous other products. The original machine trims to 240 in. and produces the heavier containerboard, used primarily in cartons and other packaging. Wood chips from waste wood are supplied from Georgia-Pacific plywood and lumber plants in Oregon.

To prevent stream pollution, Georgia-Pacific has spent over $1 million constructing two $7\frac{1}{2}$-mile long pipelines to carry plant effluent over 100 ft. out into the Pacific Ocean.

Georgia-Pacific's two kraft machines in its new Toledo mill. Number 1, at right, produces 400 tons of containerboard per day, in 72-in. diam. rolls weighing 12 tons each. Number 2 machine, at left, has a daily output of 200 tons of kraft paper, used for bagging and wrapping

Engineering for the expansion was by Stone & Webster of Boston. G-P's Toledo installation is directed by W. J. Shelton, vice-president and resident manager. President of the paper company is Robert E. Flowerree, Portland.

Hammermill Paper Co.

Oakes to Manage Oswego Division

Victor S. Oakes has been appointed division manager of the Oswego Division of Hammermill Paper Co. at Oswego, N. Y. He will be responsible for management of the entire Oswego Division when Hammermill takes title and possession of the property late this fall.

Howard Paper Mills, Inc.

Minch Elected President

John E. Minch, Sr., has been elected president of Howard Paper Mills, Inc., a subsidiary of the St. Regis Paper Co. He succeeds W. B. Zimmerman, who was drowned in a boating accident July 20.

At the same time, the Howard board of directors named Archibald Carswell, executive vice-president of St. Regis.

Mr. Minch, who is a native of Middletown, Ohio, was formerly vice-president, director of sales for Howard. He is a member of the firm's board of directors and executive committee.

The Mead Corp.

Company to Continue Stream Improvement Project at University of North Carolina

The Mead research project on the treatment of liquid waste from a semichemical pulping process at the School of Public Health, University of North Carolina, will be continued, it was recently announced by H. E. Whitaker, chairman of the board, The Mead Corp.

The project, initiated June 1, 1958, was established with the objective of discovering additional approaches to the solution of the color pollution problem. Dr. James C. Lamb, III, Associate Professor of Sanitary Engineering at the School of Public Health, has been the director and principal investigator of the project.

The continuation of the Mead project is based on the progress made by the University during the past two years and on the company's continuing determination to solve the Tuckaseegee River Pollution problem in North Carolina.

Nekoosa-Edwards Paper Co.

O'Brien Named Manager of Manufacturing at Potsdam

John O'Brien has been named manager of manufacturing for the Potsdam mill of Nekoosa-Edwards. He will be responsible for all paper machines, finishing and shipping operations as well as production and quality control.

Recently the responsibilities of Charles Sigvardt, manager of manufacturing operations for the firm's Wisconsin mills, were extended to operation at the Potsdam mill.

Oxford Paper Co.

Timberlands Sold to Nova Scotia

Oxford has sold to the Province of Nova Scotia, for approximately $3,750,000, a total of 396,000 acres of leasehold and 12,000 acres of freehold timberlands at Cape Breton, N. S. As a result of the acquisition in 1955 of a large block of timberlands in the State of Maine, William H. Chisholm, president of Oxford, deemed the Nova Scotia properties to be no longer required.

Riegel Paper Corp.

Expansion of Research Laboratory at Milford

Work is now in progress to expand to Riegel Research

Laboratory at the Milford, N. J., mill. According to D. G. Magill, technical director, most of the new area will be devoted to new test equipment, chemistry laboratories, and expanded pilot plant facilities for customer service.

Facilities for testing packaging materials are being enlarged to accommodate increased research on current problems related especially to food, or food packaging. This will include installation of automatic packaging machinery which will be used in conjunction with development of web machinability as well as product protection. In this way, the Riegel Laboratory will be in a position to solve customer packaging problems from product to final package.

Scott Paper Co.

$3 Million Expansion of Nonpaper Products

Scott announces that it will invest more than $3,000,000 in an expansion program centered on the construction of a new plant to make 'Scottfoam" and plastic cups. Due to begin soon, completion is scheduled for early 1961. The new facility will cover about six acres of land fronting on the Delaware River in Eddystone, Pa. The factory site is part of a 63-acre tract that Scott has agreed to buy, subject to formal conveyance, from Sun Shipbuilding and Drydock Co. United Engineers and Constructors designed the new building and will supervise its construction. Scott engineers worked with United on the plans for the factory, and also designed the equipment for it.

Promotions

Scott has announced three promotions in production management affecting personnel at plants in Pennsylvania, Washington, and Mexico.

Loren V. Forman, general manager of Scott's West Coast Division, will move to staff headquarters in Chester, Pa., where he will serve as executive assistant to the vice-president of production, engineering, and research.

Mr. Forman will be succeeded by Roy L. Davies, executive vice-president and director of Compania Industrial de San Cristobal, S.A., a Scott affiliate in Mexico.

Henry S. Godshall, Jr., currently manager of Compania Industrial de San Cristobal, will succeed Mr. Davis.

Thilmany Pulp & Paper Co.

MURCO Chipper Movie

A 16 mm., optical sound color movie of the latest MURCO 15-knife 110-in. diam. disk pulpwood chipper in operation in the new Woodroom at Thilmany Pulp & Paper Co., Kaukauna, Wis., has been produced by the manufacturer, D. J. Murray Mfg. Co., Wausau, Wis.

Of particular interest to woodroom operators is the unusual action of small diameter logs in the MURCO V-type spout. The film is available for showing by appointment. Write the company, Pulp and Paper Mill Sales Division at Wausau.

Educational Institutions

The Institute of Paper Chemistry

Fifth Industry Seminar

The Fifth Industry Seminar, June 15–July 7, attended by 22 representatives of pulp and paper companies throughout the United States, was recently concluded at The Institute of Paper Chemistry. The three and one-half week program, directed by Dr. A. Neil McLeod, Institute Economist, was designed to promote an increased awareness of the contributions to be made by science and technology to the industry and to broaden the outlooks of the men in attendance. Consideration was given to the impact of geographic, economic, political, and social factors which have brought the industry to its present state of development.

Attending the Fifth Industry Seminar, are, seated: J. G. Strange, IPC; O. R. Hurd, International Paper Co.; C. Schenker, Buckeye Cellulose Corp.; R. R. Chase, Union Bag-Camp Paper Corp.; W. C. Chapman, Union Bag-Camp Paper Corp.; O. A. Marrow, Riegel Paper Corp.; W. J. Bull, Champion Paper & Fibre Co.; W. R. Owens, N. Carolina Pulp Co.; M. B. Cunningham, Sonoco Prods. Co. Second Row: A. N. McLeod, Seminar Director; G. D. Jernegan, IPC; J. Noble, Olin Mathieson Chemical Corp.; R. A. Paramore, Packaging Corp. of America; R. E. Bowyer, Chesapeake Corp.; W. M. Buedingen, Owens-Illinois; J. R. Carney, Oxford Paper Co.; T. W. Orr, Marathon Southern Corp.; W. A. Thomas, Mead Corp. Third Row: J. G. Meiler, Bowaters Eng. & Dev. Inc.; D. M. Ferguson, St. Regis Paper Co.; N. H. Anderson, Longview Fibre Co.; P. F. Marsden, Scott Paper Co.; A. E. Dentremont, Great Northern Paper Co.; D. L. Wolfe, Kimberly-Clark Corp. Not pictured: K. O. Wassen, West Virginia Pulp & Paper Co.

Western Michigan University

Twenty-Five Complete Second Coating Course

Twenty-five representatives of the paper industry and of suppliers to the paper industry completed the second session

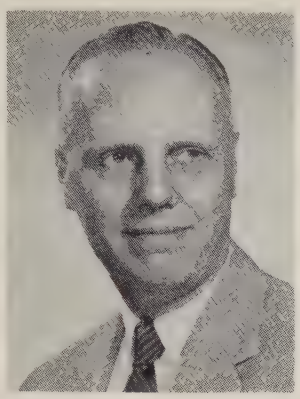
Attending the course at WMU are, front row, left to right: R. P. Tidman, Scott Paper Co.; Peyton Wheeler, Minerals and Chemicals Corp. of America; William Welsh, Gulf States Paper Corp.; Erbest M. Cook, Black Clawson Co.; Aldo Venturini, A. B. Dick Co.; Paul Hoelderle, Watervliet Paper Co.; Edward Nissen, Union Starch & Refining Co.; Second row: Dr. John R. Fanselow, WMU faculty; Eugene F. Paschall, Corn Products Co.; E. C. Garver, Armour Industrial Chemical Co.; C. Duane Coates, Mead Corp.; Edward M. Lavery, Anheuser-Busch, Inc.; Howard L. Peper, The Hubinger Co. Third row: Milton Hurston, Kimberly-Clark Corp.; William T. Goward, Clinton Corn Processing Co.; Frederick J. Tasker, Howard Smith Paper Mills, Ltd.; William Burr, Jr., Dewey & Almy Chemical division, W. R. Grace & Co.; B. S. H. Hatfield, Jonquiere Paperboard Mill, Price Bros. & Co., Ltd.; Fourth row: Raymond L. Janes, WMU faculty; John E. Maryanski, Corn Products Co.; William T. Craig, Canada Starch Co., Ltd.; Dr. A. H. Nadelman, WMU faculty; Charles D. Ingram, Scott Paper Co.; Fifth row: William Holtzman, Glidden Co.; Gerald Chambliss, Alton Box Board Co.; Jack L. Decker, Columbia River Paper Co.; William F. Nye, Jr., The Hubinger Co.; Sixth row: Dr. R. A. Diehm, WMU faculty; James Gambrell, International Paper Co.

of the annual course, "Principles and Practice of Coated Paper Manufacture," offered each summer in two, three-week classes at WMU.

North Carolina State College

Landes Joins School of Forestry

Chester G. Landes, chemical consultant of Charlotte, N. C., has been appointed Assistant Professor of Pulp and Paper Technology in the School of Forestry, at North Carolina State College, Raleigh.



Chester G. Landes

Professor Landes is the author of a number of technical articles and holds over 25 United States patents in the paper and chemical fields. His industrial background includes over 30 years research and production experience with Mead Corp., Fitchburg Paper Co., American Cyanamid Co., and Wica Chemicals, Inc.

He is a member of TAPPI and is vice-chairman of its Coating Committee. He is also a member of the American Chemical Society, the Technical Association of the Graphic Arts, the American Association of Textile Chemists and Colorists, and the Chemists Club of New York.

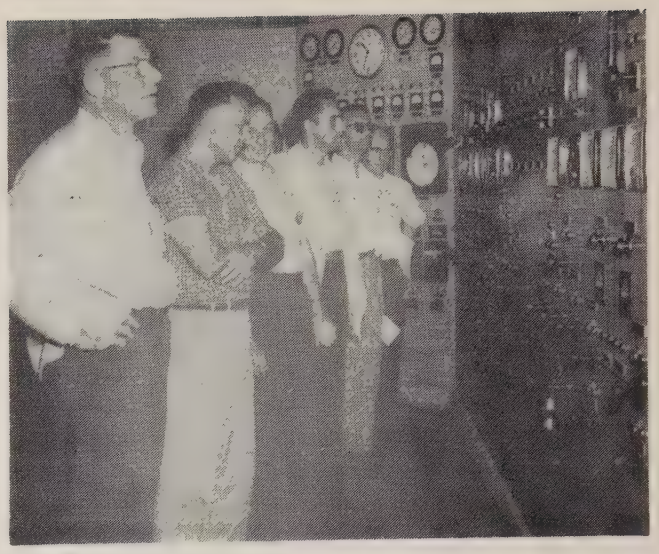
Professor Landes will continue to maintain a consulting office in Charlotte where he is a member of the Newell Toastmasters Club and the Carmel Country Club.

University of Maine

Frank A. Knight, center, division manager of the Eastern Fine Paper and Pulp Division of the Standard Packaging Corp., is shown presenting a check for $1000 to Ralph A. Wilkins, treasurer of the University of Maine Pulp and Paper Foundation. Watching at left is J. Larcom Ober, president of the foundation. Wilkins is president of Bird and Son, Inc., of East Walpole, Mass. The $1000 will go into the foundation's scholarship fund which aids deserving University of Maine students studying pulp and paper technology

Industry personnel attending the University of Maine's summer institute for paper technology, August 1–22, were: first row, left to right: Maurice Asselin, St. Regis Paper Co.; William Dow, John A. Manning Paper Co.; Harvey Haber, U. S., Playing Card Co.; Lyle Jenness, professor and head, Department of Chemical Engineering, U. of Maine; Clayton Bockus, associate professor of pulp and paper technology, U. of Maine; Andrew Chase, associate professor of chemical engineering, U. of Maine; Kenneth Thatcher, International, Paper Co.; Robert Thibadeau, Thiele Kaolin Co.; Edward Strazdins, American Cyanamid Co.; and Richard Santa Marie, Midland-Ross Corp. Second row, left to right: Gordon Inskeep, St. Regis Paper Co.; Joe Fleming, Jr., Fleming & Sons, Inc.; Lee Johnson, Monsanto Chemical Co.; H. Cummings, Monsanto Chemical Co.; Daniel Folsom, Monsanto Chemical Co.; Allen Hold, St. Croix Paper Co.; Alfred Anish, Crane Co.; Frederick Witek, Chemical Fine Paper & Bd. Viv. Std. Pkg. Corp. Third row, left to right: Frank Basile, General Aniline & Film Corp.; A. Olszewski, Monsanto Chemical Co.; John Johnstone, Spaulding Fibre Co., Inc.; Mendel Rast, United States Gypsum; Rodney Charles, Spaulding Fibre Co., Inc.; Gregg Doll, Doll Paper Co.; Russell Craig, Hercules Powder Co.; John Winship, St. Croix Paper Co.; Maurice Burnston, TAPPI; Philip Askman, Chas. T. Main, Inc.; Douglass Grant, Penick & Ford Ltd.; Vincent Doiron, International Paper Co.; Howard Snow, J. & J. Rogers Co.; and Willard Blackney, Jr., The Dow Chemical Co. Fourth row, left to right: W. Ayers, Rice Barton Corp.; Charles Babb, Knox Woolen Co.; Hammond Robertson, Jr., Finch Fruyn & Co., Inc.; William Credicott, International Paper Co.; Earle Andrews, Jr., Spaulding Fibre Co., Inc.; Guy Mitchell, Atlas Refinery, Inc.; Jack Diltz, Beloit Iron Works; Karl Dornish, Jr., S. D. Warren Co.; Carl Fishman, Beloit Iron Works; Louis Rotar, Great Northern Paper Co.; John Santoro, Newport Industries Co.; John Hosmer, Corn Products Sales Co.; Hugh Loud, Beloit Iron Works; Christopher Crane, Crane & Co., Inc.; John Gersic, Mobil Oil Co.; George McGee, The C. B. Eddy Co.; Morton McDonald, International Paper Co.; George Earley, Continental Can Co., Inc.; Bill Furcht, American Cyanamid Co.; and Richard Toothill, Hercules Powder Co. Fifth row, left to right: William Willingham, General Aniline & Film Corp.; Robert Lowney, Container Corp of America; Jenness Buck, Eastern Div. Std. Pkg. Corp.; Richard Pietro, Mohawk Paper Mills, Inc.; William Nelson, Hercules Powder Co.; John Lynch, Corn Products Sales Co.; Ronald Smith, Hercules Powder Co.; Charles Wright, The Glidden Co.; Bernard Hofreiter, N. Utilization Research & Dev. Division; Robert Durand, International Paper Co.; William Rollins, Corn Products Sales Co.; Earl Packard, Chemical Paper Co.; John Dierks, American Cyanamid Co.; James Wade, Jr., Eastman Kodak Co.; and Gerrit Klop American Cyanamid Co. Sixth row, left to right: A. Gregory, Riegel Paper Corp.; Dave Jenkins, Corn Products Sales Co.; William Nelson, U. S. Paper Mills, Inc.; Billy Moore, Fleming & Sons, Inc.; John Seydel, Seydel-Woolley & Co. Also attending the course were Thomas Calhoun and Donald Walden, both of New York & Pennsylvania Co., Inc.

Pulp technology students in the screen room of the Great Northern Paper Co., East Millinocket, Me.



Paper technology students on tour through the chemical plant, mill no. 2, Great Northern Paper Co., East Millinocket, Me.

Industry Suppliers

Albany Felt Co.

Company Holds Quality Control Panel

Achievement of highest quality in the manufacture of papermaking felts was the aim of a series of five panel discussions attended by Albany Felt Co. manufacturing personnel. More than 25 mill foremen attended the sessions at the company's Albany, N. Y., plant and later at the U. S. Gypsum Co., Clark, N. J., plant for field studies of felts in action. Gypsum's works manager, Sam Mantz, was host to the felt makers.

Felt quality is discussed by paper mill operators and Albany Felt Co. specialists. Seated left to right are Judson Hannigan, assistant manager, International Paper Co., Corinth, N. Y.; Theodore Brown, assistant superintendent, Marinette Paper Co., Fort Edward, N. Y.; Charles Benoit. manager, Strathmore Paper Co., Woronoco, Mass.; and James E. Smith, sales manager, Albany Felt Co. Standing are: Russell L. Banta, quality control; Perry H. Brown, Quality Control Director; Z. J. Chagnon, vice-president sales, and Harold R. Kleinhans, Director of Training; all of Albany Felt Co.

American Cyanamid Co.

Appointments

R. W. Lull has been appointed manager of technical service and F. W. Miner manager of sales development of the company's paper chemicals department.

Mr. Lull joined Cyanamid's paper chemicals department in 1949 and has held various technical service and sales posts at the company's Stamford research laboratories, at New York headquarters, and most recently in the Northwest.

Mr. Miner joined Cyanamid in 1947 as a research chemist. He has served in a variety of research development posts at the company's Stamford laboratories as well as technical sales and service assignments in the market development and petrochemicals departments.

American Potash and Chemical Corp.

Appointment

William W. Young has been appointed southern area regional sales manager for American Potash. He will open the company's first regional headquarters in Atlanta in the near future.

Niven D. Morgan, Jr., formerly sales representative at Shreveport, replaces Mr. Young as district manager at that office.

C. L. Straughan continues as manager of the Atlanta district and will assist Mr. Young with regional matters.

Arnold, Hoffman & Co., Inc.

Appointment

George E. Motz has been appointed to the sales staff. He will service pulp and paper accounts in the south-eastern states, working from the company's Atlanta sales office.

During the 14 years prior to this appointment Mr. Motz was sales and technical representative for Nopco Chemical Co. He is a member of TAPPI, including the Gulf Section and Southeastern Section; Salesmen's Society to Dixie Pulp and Paper Mills; American Association of Textile Chemists and Colorists; associate member, Southern Paint and Varnish Production Club; American Chemical Society; and the Armed Forces Chemical Association.

George Motz

Atlas Mineral Products Co.

Chemical Linings and Atlas Merge

Effective August 22, Chemical Linings, Inc., Watertown,

N. Y., will be merged with Atlas and moved from 156 Stone St., Watertown, to Mertztown, Pa., home of Atlas, where it will operate as an Atlas Division. Both Atlas and Chemical Linings are now wholly owned subsidiaries of The Electric Storage Battery Co. of Philadelphia, Pa.

Chemical Linings designs and constructs corrosionproof processing vessels and structures for the pulp and paper industry and the processing industries. Atlas, the country's largest manufacturer of corrosionproof cements and a leader in the field of corrosion control since 1892, has, for some time, been manufacturing materials for Chemical Linings.

The Bauer Bros. Co.

Centri·Cleaner Specifications and Application Data

Bulletin P-4-E, covering the Bauer Centri·Cleaner line, has been completely revised and reprinted. The new literature includes data on a new 180 g.p.m. cleaner, the No. 609-9″ for use on fine papers. Also included are data on head diameter, pressure drop, capacity, inlet and outlet pipe diameter, etc., for all cleaners Bauer supplies. Ask for Bulletin P-4-E, from The Bauer Bros. Co., Springfield, Ohio.

Bulletin on Product Lineup

The complete line of Bauer equipment for the pulp and paper industry is described in capsule form in Bulletin "P," now available from the Company in Springfield, Ohio.

Included are photos, specifications, and typical uses of Bauer refiners, Centri·Cleaners, Pressafiners, digesters and auxiliary equipment. Functions of the company's Plant Project Division and Laboratory Facilities are also described.

Becco Chemical Division, Food Machinery and Chemical Corp.

Offices Move to Manhattan

The administrative and sales headquarters of Becco, including Dewey H. Nelson, Albert P. Shutts, and John R. Hopkins are now located at 161 East 42nd St., New York 17, N. Y. For the immediate future continue to place your orders as before. All technical and sales information matters, however, should be directed to the new location.

Betz Laboratories, Inc.,

Steam Purity Measurement

A new water conditioning data sheet entitled, "Newest Approach to Steam Purity Measurement," has been published by Betz Laboratories, Inc., consultants on industrial water problems.

The data sheet discusses how the determination of the sodium content of steam has unveiled the causes of turbine and superheater deposits that heretofore have gone undetected.

Copies of this data sheet are available to the readers of this publication by writing to Betz Laboratories, Inc., Gillingham and Worth Sts., Philadelphia 24, Pa.

The Black-Clawson Co.

Company to Equip Menasha Mill

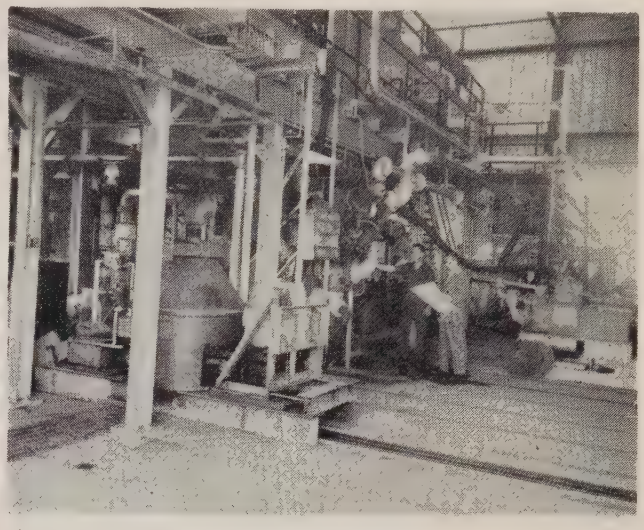
Menasha Wooden Ware Corp. has awarded contracts to Black-Clawson for the construction of the principal items of equipment to be installed in the new Menasha Pulp and Paper Division mill now under construction at North Bend, Ore. Pulping and papermaking machines from three Black-Clawson divisions will equip the mill for integrated production of 125 tons per day of 0.009 corrugating medium from Douglas-fir and western hardwoods.

For neutral sulfite semichemical pulping of the wood chips, the Pandia Division is supplying a two-tube Pandia continuous digester system including screw feeder and continuous discharger. The mill's wood supply will consist of approximately 75% alder and 25% fir chips from the lumber and plywood plant operated by Menasha Wooden Ware in the North Bend area.

E. C. Manders, manager of the Menasha Pulp and Paper Division, reports that the mill will be completed and in production by June, 1961.

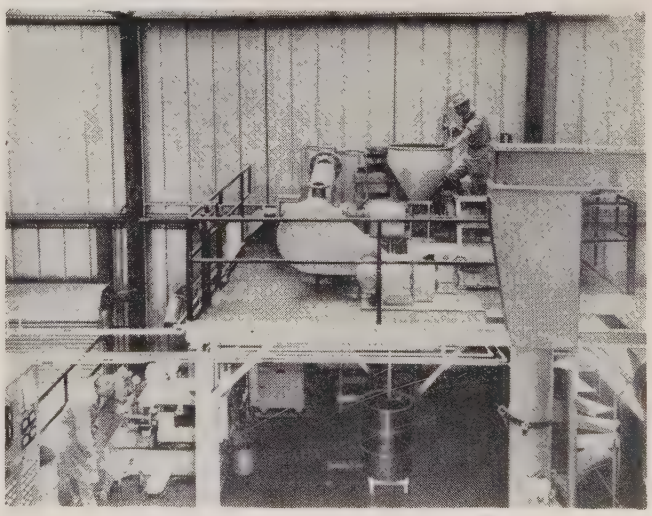
New Research Center

The construction of a new pulping and papermaking re-



A variety of pilot stock preparation equipment permits simultaneous scheduling of several test programs

The testing laboratory



The pilot, cold soda pulping system

search and development laboratory has been completed by the company in Middletown, Ohio. Devoted to advancements in the Black-Clawson equipment line and to customer service projects, the research center is housed on the site of Black-Clawson's Shartle Division.

The facility includes: complete wet and dry testing stock preparation laboratories and pilot plant, expanded and moved from its original location in Fulton, N. Y.; a new Pandia Chem Preg cold soda pulping system pilot plant; and special equipment for testing new papermaking techniques.

Stock preparation facilities. The layout of pilot equipment for pulping, cleaning, screening, and refining paper stock is designed to permit duplication of a variety of stock preparation procedures. Pilot-scale units of such standard Black-Clawson equipment as the Hydrapulper, Selectifier screen, Liquid Cyclone, Sutherland refiner, jordans, breaker trap, and pumps, form the basis of the stock preparation system. In addition, there are newly designed machines in various stages of development.

Pandia Chem Preg cold soda system. One of the businest installations in the Research Center is a complete pilot plant capable of producing up to one ton per day of cold soda pulp by the new Pandia Chem Preg System.

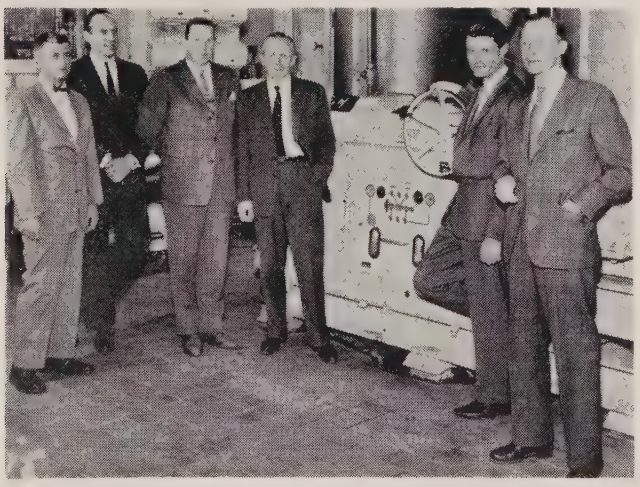
Available to all who wish to evaluate cold soda pulping, the pilot plant has recently made successful demonstration runs on such wood species as aspen, oak, red alder, and mixed hardwoods.

R & D organization. The research center is operated by the Research and Development Department of Black-Clawson under Research Director Joe Baxter, Jr. H. P. Espenmiller is director of the research center. In addition to development work in pulping and stock preparation for the benefit of the Pandia and Shartle Divisions, the center also carries out certain research projects for the Paper Machine Division, Watertown, N. Y.

John W. Bolton & Sons, Inc., The Emerson Manufacturing Co., Division.

Company to Represent Raffinator Disk Refiner in North America

Officials of John W. Bolton & Sons, Inc., and The Emerson Manufacturing Co., Division, Lawrence, Mass., have completed arrangements with Aktiebolaget Defibrator, Stockholm, Sweden, which make the American manufacturer of stock preparation machinery the exclusive sales representatives in the United States and Canada for the Swedish built Raffinator disk refiner.



Officials of Bolton-Emerson and Aktiebolaget Defibrator confer in Stockholm prior to the American firm's appointment as exclusive sales representative in the U. S. and Canada for the Raffinator disk refiner. From left to right: A. L. Bolton, Jr., treasurer and general manager, John W. Bolton & Sons, Inc.; John Mladota, president of Bolton-Emerson S. A., Lausanne, Switzerland, the firm's overseas subsidiary; Stig Selander, vice-president; Arne Asplund, president; Rolf Reinhall, chief engineer; and, Olav Naslund, general manager, all of Aktiebolaget Defibrator

Available for the first time on this continent, the Raffinator disk refiner has had wide acceptance in European paper mills where nearly 700 installations have been made. It is noted for its precision and ruggedness of construction and its capacity for performing all types of defibrating and pump through or pressure refining at high consistency. Unusual features of this disk-type refiner are its complete hydraulic system, its its visual indicator, magnified 80 times; and its ability to operate with plates in a fixed position or on a floating principle.

Full information on the Raffinator will be furnished on request to the company.

Literature on the Claflin 303

A bulletin describing the Emerson Claflin 303 continuous conical refiner for pulp and paper mills available from Emerson.

Highlighted by engineering drawings, sectional photographs and descriptive text, the bulletin treats the many performance features of this newly re-designed refiner. Contained is a list of furnishes which are being refined successfully by the many hundreds of Claflins now in use in pulp and paper mills. It further illustrates the refining capacity range of each furnish and gives full information on Claflin specifications and the types of fillings available.

A copy of Bulletin No. C-560 will be sent on request. Write to Bolton-Emerson, Lawrence, Mass., or call MUrdock 6-6171.

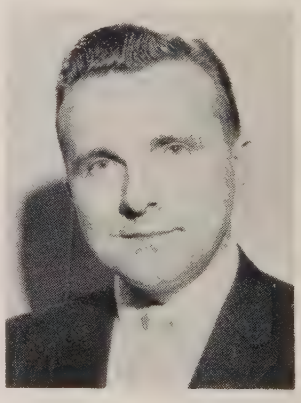
Joann Graham, R. L. Wilham, Bolton Award Winners

Mrs. Joann Graham, secretary, central personnel department, Packaging Corp. of America, Hutchinson, Kan., and R. L. Witham, plant guard at Great Northern Paper Co., East Millinocket, Me., received each a $200 check and a hand-engrossed wall plaque for their part in winning the Bolton Award essay competition. Ten cash prizes are awarded every year in this contest; the subject this year was "Through the Grapevine."

Appointment

William E. Gundlach has been appointed sales representative for the firm's full line of quality machine knives in the territory including Ohio and Western Pennsylvania.

Prior to joining Bolton, Mr. Gundlach worked for the Wheeling Corrugating Co., a subsidiary of Wheeling Steel Corp., the last three years as sales manager in the Pennsylvania territory.



William E. Gundlach

The Borden Chemical Co.

Corrugated Container Adhesive

A 10-page report which describes technical specifications for corrugated container glue-lap production and includes a review of synthetic resin emulsion glues designed specifically for this purpose is being offered by Borden.

For a free copy write to Department H, The Borden Chemical Co., 350 Madison Ave., New York 17, N. Y.

Appointment

Alvin E. Jerome has been appointed sales representative for the Polyco-Monomer Dept. of Borden, a division of The Borden Co.

Mr. Jerome will be responsible for the sale of Polyco-Monomer's synthetic resins in the New England states.

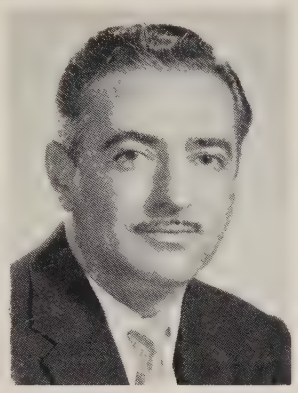
Cameron Machine International AG

Subsidiary Companies Established

Cameron Machine Co., Dover, N. J., manufacturers of slitting and roll winding machinery have announced the formation of subsidiary companies to further the international marketing of their equipment.

Cameron Machine International AG, a wholly owned subsidiary of Cameron Machine Co., will be the parent company for other subsidiary companies including Cameron Maquinas Ltda., Sao Paulo, Brazil, and Cameron Europe S.A., Paris France.

Founded in Zug, Switzerland, the purpose of Cameron Machine International will be to serve as a sales and service company, in all areas not covered directly by other Cameron companies, for the Cameron, Clark-Aiken and Tidland Machine Co.'s equipment. It will also extend rights through licensing agreements for manufacture outside the United States and Canada.



Guillermo Velez

Guillermo Velez, associated with Cameron's export sales for over ten years has been appointed sales manager for the Swiss company. Mr. Velez will continue to operate from Cameron's Dover, N. J., office.

Cameron Europe S.A., 5 Rue de Prony, Paris, France, was established for sales and service to countries within the European Common Market. The company will be headed by Robert Mace as general manager and Michel Sirvin will be the sales manager. Both men have had long associations with the paper industry in France. In addition to Cameron slitting and roll winding machinery, the French company will handle the Clark-Aiken Co.'s complete line of cutter-layboy and related finishing room handling equipment and Tidland pneumatic shafts.

Cameron Maquinas Ltda., Sao Paulo, Brazil, now in operation, is headed by Paul Chessare formerly a divisional sales manager of Cameron Machine Co. The Brazilian company is now producing Cameron's small converting machines and has plans to manufacture Clark-Aiken cutter-layboys as well as Tidland pneumatic shafts.

Chain Belt Co.

Rex Rotary Table Feeder Bulletin

Publication of an 8-page bulletin, 6093, describing its standard Rex Rotary Table Feeders has been announced by the conveyor division, Chain Belt Co., Milwaukee, Wis.

Rex rotary feeders are designed for controlled feeding of sand, crushed ore, wood chips, and similar bulk materials providing an efficient method of metering material from vertical storage bins. They also allow constant, accurate control of the quantity of material fed onto conveyors and processing equipment, thus eliminating starving and flooding.

For your free copy of bulletin 6093, contact your nearest Chain Belt distributor or district office, or write directly to the sales promotion department, Chain Belt Co., Milwaukee 1, Wis.

Chemical Concentrates

Seculate Prevents Corrosion

A Seculate coating, which dries to a rock-hard, off-white film, prevents condensation by a two-way action: surface insulations; and absorption of small amounts of moisture followed by release as the humidity drops. The material can be applied to practically any metal or building material by brushing or spraying.

Seculate has been proved in hospital sterilization rooms, in storage areas on docks, in textile and paper mills, on the walls of cold storage rooms, in various marine uses, on water mains in hydroelectric plants, in air locks, on the interior walls of switch gears cabinets, and in other applications.

For full information write to Chemical Concentrates, Fort Washington, Pa., for Bulletin 1.

Chemical Linings, Inc.

On Monday, August 22, Chemical Linings relocated at Mertztown, Pa., as a division of the Atlas Mineral Products Co.

Since last November, when the company was purchased by The Electric Storage Battery Co., of Philadelphia, it has been working closely with Atlas, another member of the ESB family. Atlas, the country's largest manufacturer of corrosionproof cements and a leader in the field of corrosion-control since 1892, has been manufacturing cements for the company for some time.

Consolidated Water Power & Paper Co.

Appointments

Two executive appointments at Consolidated Water Power & Paper Co. are:

M. V. Molsberry, chief engineer, to manager of power and engineering consultant, succeeding the late W. F. Thiele; A. R. Dent has been named chief engineer to succeed Mr. Molsberry.

Corn Products Sales Co.

Snyder Named Manager of Sales-Service

E. Carl Snyder has been appointed manager of sales service for the Industrial Division, Corn Products Sales Co. He succeeds Thomas A. Bruce who has returned to the West Coast as assistant western regional manager of the Industrial Division.

Crown Zellerbach Corp.

Hearon Vice-President of Research and Development

Crown Zellerbach's board of directors has approved a major realignment of the corporation's research functions, and has elected Dr. W. M. Hearon to the new post of vice-president for research and development.

Dr. Hearon will move to corporate headquarters in San Francisco, where he will report directly to senior officers of the company. He will continue to retain his present responsibility for the operation of the Chemical Products Division.

As part of the reorganization of the corporation's research activity, John S. Barton has been promoted to director of research in charge of the Central Research Division in Camas, Wash. Dr. Barton has been director of packaging research and development for the company's Western-Waxide Division in San Leandro, Calif. He will be succeeded by E. G. Tonn, who has been assistant director of packaging research and development.

E. H. Nunn has been promoted to assistant general manager of the Chemical Products Division.

R. R. Donnelley & Sons, Co.

Promotions

Alex Glassman, supervisor materials inspection, has announced the promotion of R. H. Stewart, K. A. Jont, and G. L. Swope to supervisors, paper inspection control.

Mr. Stewart will be responsible for the control of paper used in printing *Life*, *Time*, and *Sports Illustrated*.

Alex Glassman

Ray Stewart

Mr. Jont, paper for *Look* magazine; Sears Roebuck catalogues; and the telephone books.

K. A. Jont

G. L. Swope

Mr. Swope will be responsible for the paper for *National Geographic; Farm Journal;* and all other paper purchased by R. R. Donnelley.

Mr. Glassman is a member of the Graphic Arts Committee of TAPPI and is taking a leading part as chairman of the surface strength subcommittee.

Dorr-Oliver, Inc.

Brochure on ClO_2 Bleaching Tower

A new four-page brochure, Bulletin No. 3307, describes the new concentric bleaching tower design recently introduced by Dorr-Oliver, Inc., Stamford, Conn. (*Tappi* 43, No. 7: 120A (1960)). It is available free to readers of this publication.

New Assignments

A series of reassignments in the top-level organization of Dorr-Oliver effective since July 1, are as follows: William J. Fox, to the newly created post of vice-president—domestic operations; Douglas C. Reybold assumes responsibility for the research, development, legal, and patent functions; H. J. Talbot is managing director—Europe.

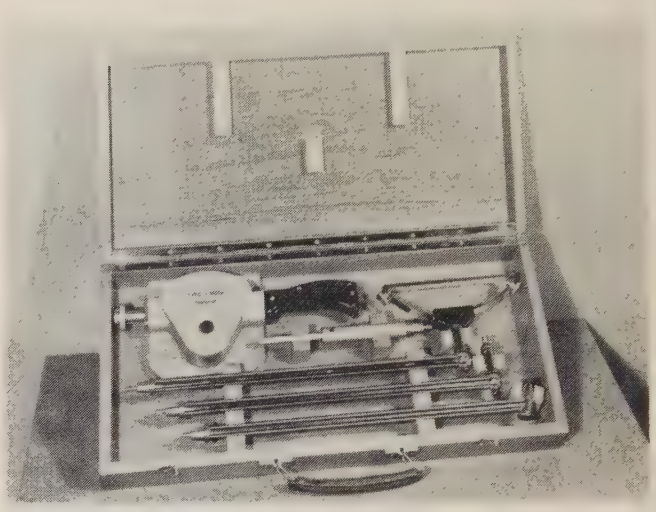
The company has also announced a number of promotions and new assignments in engineering. Harry E. Lundberg has been named director of engineering, replacing Glenn O. Wilson, who has been appointed to the newly created post of director of technical coordination; Jack S. Chasteen has replaced Lundberg as manager of filtration engineering, and Charles A. Schreiber has replaced Mr. Chasteen as manager of engineering coordination.

The general equipment engineering division has been split into two units: Russell C. Raynor heads the new sedimentation engineering division and Philip B. Booth heads the reorganized general equipment engineering division. William A. Hirschfield has replaced Booth as manager of Fluo-Solids engineering.

Epic, Inc.

Surface Contact Pyrometer

Epic's new surface contact pyrometer is self-contained and consists of a hand-held temperature indicator and a surface tracer equipped with a termocouple head. Heads are available in the form of a disk band, ribbon, or needle. All surface tracers are interchangeable and can be either rigidly or flexibly attached to the indicator. The various standard measuring ranges are available up to 1800°F. It has an automatic cold-end compensation.



The surface contact pyrometer

Fleming & Sons, Inc.

John G. Fleming, Sr., Dies

John George Fleming, Sr., 58, of 3525 Turtle Creek, chairman of the board and executive vice-president of Fleming and Sons, Inc., Dallas paper company, died Friday in his home after a long illness.

A lifelong Dallas resident, he was a graduate of the old Dallas University and attended Notre Dame University and the University of Texas.

Active in civic affairs and national and local paper associations, Mr. Fleming was a member of the National Paper Board Association and the Folding Paper Box Association. During World War II he received a citation for his contribution to industry efforts.

John G. Fleming

Freeport Sulphur Co.

Two Executives Promoted

Thomas R. Vaughan has been elected vice-president and general counsel, and John C. Carrington vice-president of sales by the board of directors of Freeport Sulphur Co.

Mr. Vaughan joined Freeport's legal department in 1942 and in 1956 was appointed general counsel for the company's nickel activities.

Mr. Carrington joined the company in 1939 as assistant to vice-president. He was made director of personnel relations in 1945 and director of public relations in 1946. In 1947 he was elected assistant to president and, in 1952, vice-president.

Graver Water Conditioning Co.

The Automatic Tape Analyzer

A 4-page, color bulletin on the Graver automatic tape analyzer is available from the company. The bulletin, WC-127, covers all facets of the unit, including method and principle of operation, potential applications and basic specifications for the instrument, and the Millipore Filter (®) tape used as the testing medium. It also gives design data, including components and materials of construction. Copies of the bulletin are available from Graver without obligation.

For further information, contact: Martin Wank, manager, sales promotion, Graver Water Conditioning Co., 216 West 14th St., New York 11, N. Y.

Goulds Pump International, Inc.

E. E. Backlund, vice-president and general sales manager of Goulds Pumps, Inc., Seneca Falls, N. Y., announced the establishment of a separate corporation, Goulds Pumps International, Inc., to handle all export sales of the company. Worldwide coverage of all important markets, excepting Continental United States, Alaska, and Canada, will be provided by the new company.

The company urges that all inquiries, correspondence, etc., covering the sale or usage of its product in foreign fields be directed to the new corporation at, 260 Madison Ave., New York 16, N. Y.

Hooker Chemical Corp.

Chemicals Digest

Ninety-four organic and inorganic chemicals produced by Hooker for industry and agriculture are reviewed in a newly revised and enlarged, 16-page, quick-reference digest, "Hooker Chemicals," recently published by the company.

Copies of the new bulletin, No. 100-D, are available to research, engineering, production, purchasing, or other interested executives on request. Write to Hooker Chemical Corporation, P. O. Box 344, Niagara Falls, N. Y.

Nonpremium Full Open-Head Steel Drum for Flake Caustic Soda Standardized

A new and improved, nonpremium, full open-head steel drum of 400–450 lb. capacity has now been made standard by the Eastern Chemical Division of Hooker.

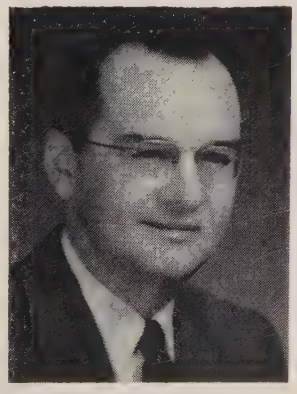
The new container replaces the company's 18-in. opening friction cap drum as a nonpremium standard package for caustic soda. The new $34^1/_2$ by 21-in., 49-gallon capacity, 24-gage drum weighs about 20 lb. It will be available for Hooker's several grades of flake caustic soda.

Huyck Felt Co.

Appointment

George H. Gill, III, has been promoted to the position of assistant sales manager. Mr. Gill was graduated from the Pulp and Paper School at Syracuse University and has worked for almost 10 years as a Huyck field service and sales engineer. He will work out of Huyck's Rensselaer, N. Y., office.

George H. Gill, III



Ralph N. Prince

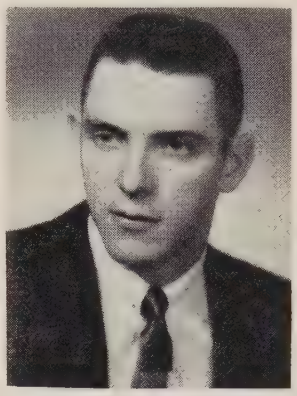
Ralph N. Prince, a veteran of almost 25 years of association with the paper industry, will replace Mr. Gill as the Huyck felt sales engineer for Ohio and portions of Indiana, Pennsylvania, West Virginia, Kentucky, and Tennessee. Mr. Prince is a chemical engineer from the University of Maine and was previously employed by Fort Orange Paper Co. and American Cyanamid Co. He will continue to make his home in Miamisburg, Ohio.

Kelco Co.

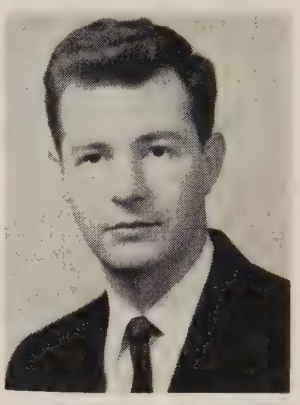
Appointment

John H. Smith has been named special representative for technical sales and service in the paper division of Kelco.

He will call on pulp and paper mills in the New England and Eastern Canada areas on behalf of Kelco Algin products, and he will be based in Chelmsford, Mass., with headquarters at the Kelco Co. paper division sales offices at 75 Terminal Ave., Clark, N. J.



Ronald B. Hough



John H. Smith

Ronald B. Hough has been appointed special representative handling technical sales and service work throughout the southern territory of the United States. He will also call on pulp and paper mills in that area on behalf of Kelco Algin products, and he will be located in the Mobile, Ala., area and work out of the Kelco Co. paper division sales office located at 75 Terminal Ave., Clark, N. J.

The M. W. Kellogg Co.

Cassell for Pulp and Paper Division

Mr. Charles W. Cassell's appointment to the pulp and paper division of The M. W. Kellogg Co. was recently announced by the company, a New York engineering and construction firm.

Mr. Cassell, an engineering graduate of Purdue University, has specialized for more than 30 years in the chemical and mechanical engineering aspects of pulp and paper mill design. He is a member of the Mechanical Engineering Committee of TAPPI, the Paper Industry Management Association, the American Society of Mechanical Engineers, and the American Chemical Society.

Before his recent appointment, Mr. Cassell directed his own consulting engineering firm, C. W. Cassell & Associates of Ann Arbor, Mich.

Appointment

Roy D. Rivers has been appointed project manager in company's pulp and paper group group.

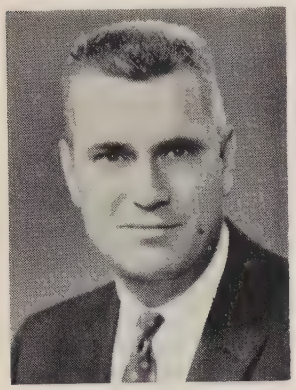
Mr. Rivers supervised startup and helped train operators at new mills located in various countries including India, Peru, and Brazil. Prior to joining M. W. Kellogg, he was mill manager for Champion Celulose, S. A., at Sao Paulo, Brazil. He is a member of TAPPI and PIMA.

KVP Sutherland Paper Co.

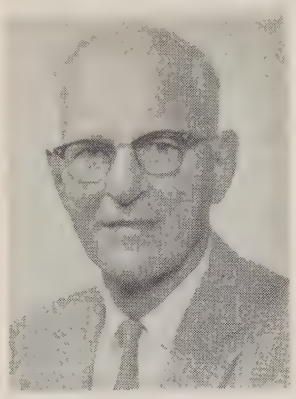
Appointments

Thomas Flanagan has been appointed technical director of the Sutherland Division and Cobe Jager, Wax Division superintendent of the KVP Division, effective September 1.

Mr. Flanagan, formerly superintendent of KVP's Wax Division, had served in this capacity since March, 1959. He had also served with the KVP Division as assistant superintendent of the Wax and Printing divisions, technical assistant to the vice president of sales, and technical assistant to the manager of manufacturing. In 1954–55, Mr.



Thomas Flanagan



Cobe Jager

Flanagan was transferred to the Research Laboratory of KVP Sutherland's Espanola, Ontario, pulp and paper mill. Prior to this, he was assigned as a laboratory chemist with the KVP Division's Research Laboratory in Kalamazoo.

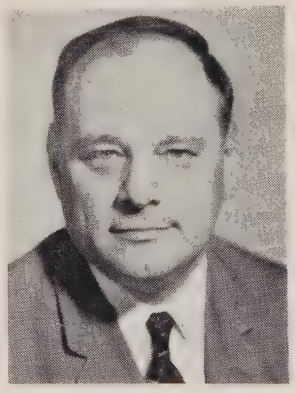
Mr. Jager was formerly superintendent of the Wax Production Department at KVP. Prior to this, he was assistant superintendent of the Wax Production Department for four years and a tour foreman in the Wax Division for six years.

Mr. Jager began employment with the KVP Division in October, 1923. During most of his nearly 37 years with the company he has been a nember of the Wax Division, serving first as a wax machine helper and then wax machine operator before his promotion to supervision.

Lodding Engineering Corp.

Appointment

William P. Dapses has been appointed the exclusive sales representative on the west coast for Lodding Engineering manufacturers of doctors, showers, fuzz removers, and other products used in the paper industry.



William P. Dapses

R. B. Mac Mullin Associates

Appointment

R. B. MacMullin Associates, chemical consultants to the paper industry and chemical process industries, announce a new partnership which includes R. B. MacMullin and P. P. Beno, present partners, and a new partner, C. A. Butler. Mr. Butler, formerly director of engineering and director of commercial development for Diamond Alkali Co. of Cleveland, Ohio, will be manager of the Associates' Cleveland office.

The Marvellum Co.

Appointment

P. C. Whiting, Jr., has taken over as Midwestern sales manager for the company. As manager of product development he was instrumental in the purchase and initial product runs of Marvellum's new polyethylene extrusion-coater.

Minerals & Chemicals Philipp Corp.

Distributor Training Conference

Service to the customer was the theme of a 3-day technical training conference recently held in Princeton, N. J., by Minerals & Chemicals Philipp Corp. for its nationwide distributor organization. Established as an annual training school by MCP's Distributor Department, the conference serves to ground its distributors in the technical phases of MCP products, bring them up to date on what is being developed in the MCP research laboratories, and call their attention to how MCP products are currently being applied in the field. This year's conference theme from MCP'S basic sales philosophy was "marketing throvgh service."

The group attending the distributor training conference, held in Princeton, N. J.

Minerals & Chemicals Philipp Corp., Menlo Park, N. J., produces a wide variety of products based on Attapulgus clay, aluminum silicate pigments, activated bauxite, lime, and limestone.

Morningstar-Paisley, Inc.

Atlanta Plant Begins Adhesives Production

Production of a complete line of industrial adhesives has begun at the new Morningstar-Paisley, Inc., plant an Atlanta, Ga. The new plant, 15th in the company's string of coast-to-coast processing facilities, will also serve as a warehousing and service facility for other Morningstar-Paisley products, such as textile, paper and food starches, water soluble gums, polyvinyl acetate resins, and plastisols.

Plant personnel will include Lee Kritzer, regional manager, and Milton F. Lenz, production manager.

Owens-Illinois

Appointment

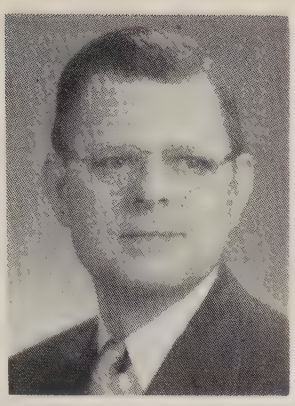
William W. Brown has been named general manager of the Owens-Illinois Paper Products Division's Milwaukee plant.

Mr. Brown, who has been assistant regional sales manager of the division's north central region with headquarters in Chicago, will be responsible for sales, administrative, and production activities at the Milwaukee plant. The plant manufactures corrugated shipping boxes.

B. F. Perkins & Sons, Inc.

Tinker Rejoins Company

Malcolm H. Tinker, Sr., rejoined the company, on August 15, as a sales engineer. For the past $6^1/_2$ years, he has been employed at National Blank Book Co. in Holyoke, first as an industrial engineer, then as director of purchases and traffic manager.



Malcolm H. Tinker

Plastics Division, Monsanto Chemical Co.

Styrene Foam Film

A flexible plastic film manufactured from styrene foam and competitive in price with paper is announced by Monsanto's Plastics Division.

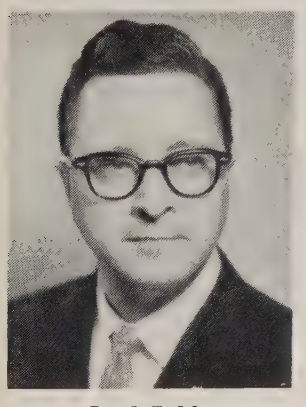
The new film, called Santofome, has the appearance and feel of kidskin, and is said to be waterproof, sanitary, lightweight, nonabrasive, and grease resistant, in addition to having good insulating and cushioning properties. It has been fully accepted for food packages by the Food & Drug Administration, according to J. P. Skehan, manager of the division's Santofome products section.

J. O. Ross Engineering Div./Midland-Ross Corp.

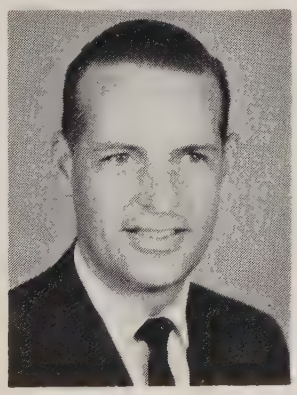
Goldner to Head Seattle Office

Paul Goldner has been appointed district manager of Seattle office.

Mr. Goldner, an experienced paper mill equipment salesman, presently covers the New York State mills from the New York office. He started with the Ross organization in 1940, after attending the City College of New York. He will succeed R. W. Grott who has been the Seattle manager



Paul Goldner



K. H. Jones

since 1956. Mr. Grott will move to the New York office of Ross, as assistant chief engineer for the division.

Jones for District Sales Manager

Kenneth H. Jones has been promoted to New York district sales manager.

His duties in the new post include the coordination and supervision of the entire sales force of the Ross New York office. This office handles sales throughout most of the Eastern United States, in many various industries.

Sandoz, Inc.

New Booklet for Paper Colorists

"Change for the Better" is the title of a new booklet just published by Sandoz.

The booklet covers the following topics:

1. Plus-Value dyes for paper.
   Each dye coded plus its Color Index Number.
2. Specialties for papermakers.
   Sandoz dyes with no prototype, and dyes with special uses.
3. Dyes for stock.
   Colors recommended for use in the beater, pulper, etc.
4. Dyes for surface application.
   Specific recommendations for calender staining, etc., with solubility limits indicated.
5. Information on paper dye performance.
   Affinity for clay, side-properties, optimum pH, etc.
6. For specific paper grades.
   Specific recommendations for specific grades.
7. For your convenience.
   A ready-reference listing of dyes used in the trade.

Copies are available to readers of this publication who write to the editor, or direct to Sandoz, Inc., New York 13, N. Y.

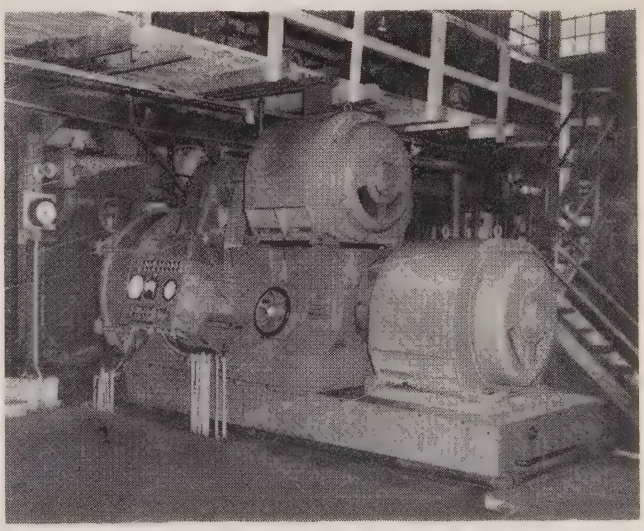
Sprout, Waldron & Co., Inc.

Ultra-Modern Testing Laboratory

A four-story building having 25,000 sq. ft. of floor space, located one block from the main manufacturing plant of Sprout, Waldron in Muncy, Pa., houses what is believed to be one of the most modern testing laboratories available to industry today.

To simulate actual in-plant conditions. The company

The new Sprout-Waldron research and testing laboratory



The new 42" double-disk refiner

has installed full-size commercial machines so that sufficient volume of material can be handled to give most accurate capacity and finished product data.

New 42-in. Double Disk Refiner

A 42-in., large capacity double-disk refiner, capable of continuous 24-hour-per-day service with 1600 applied horsepower, is now available from the company.

The first such refiner was installed at Bowaters Corner Brook, Newfoundland, mill in November, 1959, and has demonstrated superior performance and pulp quality in commercial production.

A. E. Staley Mfg. Co.,

Appointment

George A. T. Moore has been appointed assistant manager of paper sales for the company. He joined the company as an industrial salesman in 1950 and became a senior salesman in 1952. He served, successively, as assistant manager of Staley's Philadelphia and Chicago offices. In 1959 he was made administrative sales assistant of paper sales.

George A. T. Moore

Stowe-Woodward, Inc.

Nip and Crown Check of Rolls Simplified

A new and simplified method for testing both the nips and crowns of rubber covered rolls is announced by the company. The nip-width tester consists of 6-in. wide rolls of specially designed and embossed aluminum foil, with an exact thickness maintained throughout each roll of foil.

Procedure for making the test gives both fast and accurate results. A strip of foil is pulled from the dispenser, placed across the face of the rubber roll to be checked, and is fastened in place with strips of pressure-sensitive tape. Reduced pressure is applied to engage the rolls and bring ends into contact evenly. As the pressure is increased, the foil is pressed flat in the nip so that a picture is taken of the nip contact area. The sensitivity of the foil pattern provides a sharp, clear impression which permits measurements to within one hundredth of an inch. Extreme variations are immediately discernible. An example would be an end-to-end variation which could indicate uneven side-to-side loading. This might be the case if there was a malfunctioning of the loading mechanism or pressure gages. Before startup the situation could be corrected.

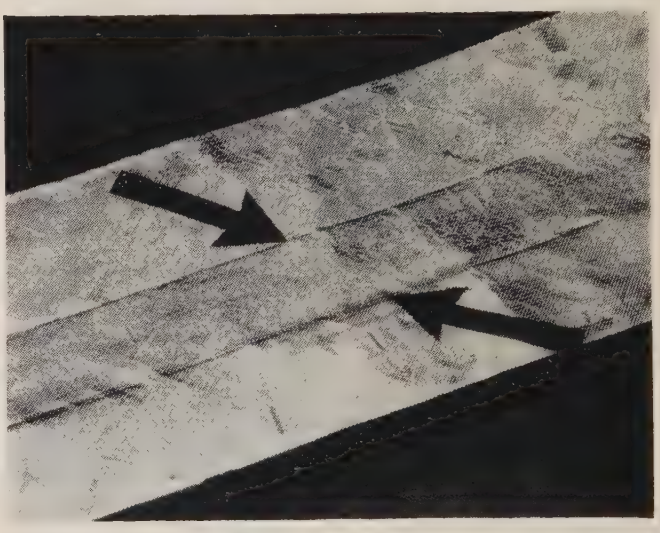
In the area of fine measurements a pair of fine point dividers and a 6-in. rule graduated in hundredths of an inch are used to measure the nip impression on the foil. Intervals of one or two inches along the pattern are marked off and a reading is taken at each point. This would compare with the divisions used to measure crown on the face. The readings are then plotted and analyzed.

The principal value of the nip-width is to help avoid troubles before they start. When a newly covered roll is placed in a machine, its crown is static, cannot be changed. The test can enable the loading pressure to be adjusted for best possible results with the existing crown.

A test before the machine is started may reveal uneven loading of one or both rolls. When an extremely wide or narrow pattern is noted on the foil at either end, steps to compensate the unevenness can be taken. Many off-standard factors can be discovered with the nip-width test since any variable can alter the effects of the nip.

Additional information from Stowe-Woodward, Inc., Newton Upper Falls, Mass.; Griffin, Ga.; or Neenah, Wis.

The nip-width tester is a metal dispenser containing a roll of 6-in. wide embossed aluminum foil. Pressure applied to rubber covered rolls when the foil is in place creates a "flattened" area on the foil, thus permitting a quick visual check of the nip contact area



Close up photo of aluminum foil after nip and crown test shows areas where roll pressure has depressed the embossed surface of the foil. Study of the strip covering the entire length of the rolls provides a quick check of both nip and roll crown

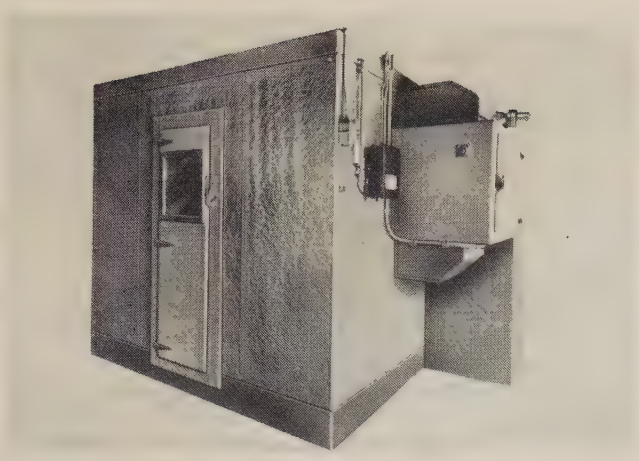
Testing Machines, Inc.

The "Uni-Lab" Controlled Temperature and Humidity Laboratory Room

TMI announces the Model TMI-100-1 "Uni-Lab," which is capable of producing and maintaining controlled temperature and humidity (within +70 to +80°F. dry bulb temperature, and 40 to 75% R.H.). The "Uni-Lab" can also be made available for an R.H. range to 90% and dry bulb temperature up to 120°F.

"Uni-Labs" are available in various sizes although the standard interior is 6 ft. wide by 6 ft. deep by 7 ft. high Approximate exterior is 7 wide by 7 deep by 9 ft. high, plus approximately 3 by 4 ft. space for two pen recorder and conditioning equipment. Larger interiors are also available.

Four further details write to Testing Machines, Inc., 72 Jericho Turnpike, Mineola, N. Y.



The Uni-Lab

TMI Curl Tester

A data sheet is now available which illustrates and describes the TMI curl tester. This tester is designed for use in determining the amount of curl which occurs in paper when the relative humidity is changed.

For complete details write to Testing Machines, Inc., 72 Jericho Turnpike, Mineola, N. Y.

Thwing-Albert Instrument Co.

Torsion Tear Tester

The Albert torsion tear tester has been developed to measure shear strength or tearing resistance of strongly directional materials difficult to measure by other methods. Reinforced shipping tape, corrugated boxboard, textiles, highly calendered rubber stocks, etc., usually tear at right angles to the direction of greatest stength.

Bulletin 265 describes the instrument and the method of operation as well as giving typical applications.

Accessories for the Elmendorf Tearing Tester

Bulletin 211-ELM describes two accessories for the Elmendorf tearing tester: the precision sample cutter, which speeds up the preparation of accurate, uniform samples; and the check weights, developed for accurate, easy, and rapid checking of the calibration of the tester.

Copies of the bulletin may be obtained from the company at Penn St. & Pulaski ave., Philadelphia 44, Pa.

Tropical Paint Co.

Booklet on Painting Announced

Selection and use of maintenance paints in the pulp and paper industry is the subject of a nine-page booklet prepared by the Tropical Paint Co. of Cleveland.

Entitled "Recommended Maintenance Painting System for Pulp & Paper Mills," the booklet describes various paint systems designed to withstand the moisture and chemical exposures of paper mills.

A free copy may be obtained by writing to the company, 1246 West 70th St., Cleveland 2, Ohio.

United Container Co.

Company Acquires Bicking Paper Mills

United Container has acquired the Bicking Paper Mills, Downingtown, Pa. The firm will continue operation as a division of United Container under the name of Bicking Paper Mills and consultation will continue to be offered by Eugene Kardon Enterprises, the management firm which arranged for the acquisition of Bicking Paper Mills by United Container.

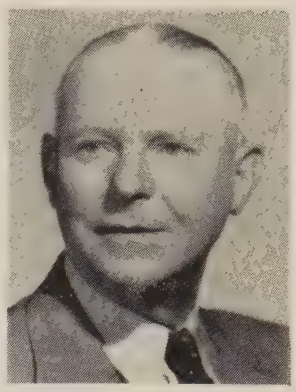
Frank J. Keating, with United Container 14 years, has been named vice-president of the Bicking Paper Mills and Donald A. Cumfer, general manager at Bicking.

Waldron-Hartig Division/Midland-Ross Corp.

Charles A. Dickhaut Retires

Charles A. Dickhaut, designer and sales engineer at Waldron-Hartig, retired Sept. 1, 1960, after 34 years of service with Waldron.

Mr. Dickhaut is best known for his contributions in the coating methods and web alignment area of the web processing field. He is the holder of 17 patents in this area.



Charles A. Dickhaut

General

FDA Clearance for PVAcetate Emulsion Sought

The first step in obtaining federal clearance of polyvinyl acetate emulsions for use in food packaging applications was taken recently by a committee made up of representatives of the nation's emulsion manufacturers.

The committee, a temporary technical organization established earlier this year, has submitted a petition requesting clearance of polyvinyl acetate emulsions by the Food and Drug Administration.

Announcement of the action was made by Harold L. Jaffe, Colton Chemical Co., a Division of Air Reduction Co. Inc., who is serving as chairman of the committee. According to Mr. Jaffe, all technical information developed by the committee will be made available to all segments of the packaging industry.

All interested manufacturers of polyvinyl acetate emulsions are invited to join the committee.

For further information, contact the secretary, David S. Cox, Product Development Dept., Shawinigan Resins Corp., Springfield, Mass.

Spent Liquor to Cut Steel Costs

Major iron ore corporations and sulfite pulp manufacturers have joined forces in a research project at Michigan College of Mining and Technology, Houghton, Mich. This study is aimed at using spent sulfite liquor to cut present costs of making steel from the low-grade iron ores of the Lake Superior region.

Quantities of spent sulfite liquor that would be required for the uses under study at Michigan Tech might approach total tonnages now otherwise unused and available from all Wisconsin and Minnesota pulp mills. According to Averill J. Wiley, technical director, Sulfite Pulp Mfr.'s Research League, results have been sufficiently encouraging so that iron ore processors are intensively exploring these possibilities in cooperation with Michigan Tech and Paper industry researchers. The League is contributing $8000 for investigation of the uses of sulfite materials in pelletizing iron ore concentrates without the costly high temperature hardening now required to prepare the low-grade ores for shipment to the blast furnaces.

An advisory committee of iron ore industry technical experts—metallurgists, chemists, and engineers—chosen by the iron companies helps guide the research project and keeps their industry informed of results. The project is under the immediate charge of professor M. E. Volin, director of the ore dressing laboratory.

IPACK Exhibition Postponed

In accordance with the wishes of almost all exhibitors the IPACK exhibition will be held June 20 to 29, 1961.

Progress in Nuclear Power

J. W. MAGEE and J. H. WRIGHT

THE Progress in Nuclear Power Subcommittee recently provided the Steam and Power Committee a year-end summary of the atomic industry as prepared by the Atomic Industrial Forum, a nonprofit membership association incorporated for the purpose of fostering the development and utilization of atomic energy for peaceful purposes. In the interests of providing *Tappi* readers a concise summary of the highlights of nuclear power development to date, it was considered that this report was worthy of reproduction:

The release follows:

"The atomic industry closed out 1959 and looked ahead to the next decade encouraged by evidence of increasing willingness of reactor manufacturers to offer guarantees of nuclear power costs and reactor performance, by a 10-yr. U. S. Atomic Energy Commission plan for attainment of economic nuclear power, and by continuing progress in construction and operation of actual nuclear power projects and supporting programs, according to a year-end summary issued by the Atomic Industrial Forum, the association of the nuclear industry.

Dresden Plant Completed. The most significant single industrial event of the year was the completion of the United States' first large privately built and owned nuclear power plant at Dresden, Ill. The 180,000 electrical kilowatt installation was built by the General Electric Co. for Commonwealth Edison Co. of Chicago, and was completed six months ahead of schedule. It uses a reactor of the boiling water type. When the plant completes its testing phases and reaches full power in mid-1960, it will be the most powerful single reactor for electricity production in operation anywhere in the world.

Power Cost Guarantees. Some examples of the willingness of manufacturers to underwrite conditionally the cost and performance of nuclear plants were the following:

J. W. MAGEE, JR., and J. H. WRIGHT, Westinghouse Electric Corp., Philadelphia, Pa.

1. Westinghouse Electric Corp. said it is ready to develop and manufacture by 1965 a 330 electrical megawatt pressurized water reactor plant to produce power at costs competitive with fossil fuels in the "higher cost areas" of the United States. The company will guarantee "all elements of cost over which it has control," defining these as plant capital cost, plant capability, and fuel life. It pointed out that power cost elements, such as plant load factor, individual annual charges, and operation and maintenance costs, are subject to the control of the operating utility and to local circumstances.

2. General Electric Co. said it has been quoting power reactors on a fixed price basis, and adds: "in a very few areas our plants show promise of being competitive in the very near future." Factors currently being guaranteed are plant output and fuel life.

3. For organic cooled and moderated, as well as sodium graphite reactor concepts, the Atomics International Division of North American Aviation, Inc., has announced that it will guarantee plant output, capital costs of the reactor, and fuel life.

4. The Babcock & Wilcox Co. has announced that it will guarantee fixed prices on plant capital costs, power output, and fuel life for pressurized water reactors.

10-yr. National Program. One of the year's most important encouragements to the United States nuclear industry was the announcement by the U. S. Atomic Energy Commission that it had established as an official objective of national policy the achievement of nuclear power costs competitive with fossil-fuel power costs in some areas of the United States within 10 years. A technical program designed to achieve this objective, with the cost and effort to be shared by both government and industry, is being prepared by the AEC.

Preliminary studies made by the AEC in connection with the 10-yr. program indicate that construction could begin today on a large (300,000 electrical kilowatt) nuclear plant of the pressurized water reactor type which could reasonably be expected to produce power at a cost of 9.28 mills per kilowatt hour. The studies also indicate that, based on antici-

U. S. Power Developments—1959

Owner/operation (designation, if any)	*Location*	*Type*	*Output, electrical megawatts*	*Principal contractor*
Completed				
Commonwealth Edison Co.	Dresden, Ill.	Boiling water	180	General Electric Co.
Began construction				
AEC—Experimental Gas Cooled Reactor (EGCR)	Oak Ridge, Tenn.	Gas cooled, graphite moderated	25	H. K. Ferguson Co.
AEC/City of Piqua	Piqua, Ohio	Organic moderated	11.4	Atomics International
U. S. Army Corps of Engineers—Portable Medium Power Plant No. 2A (PM-2A)	Greenland	Pressurized water	1.5	Alco Products, Inc.
Contracts signed				
Consumers Power Co.	Big Rock Point, Mich.	Boiling water	50	General Electric Co.
Philadelphia Electric Co. and High Temperature Reactor Development Associates (HTRDA)	Peach Bottom, Pa.	Gas cooled	28.5	Bechtel Corp.
Carolinas Virginia Nuclear Power Associates	Parr Shoals, S. C.	Pressurized water	17	Westinghouse Electric Corp.
Plans announced				
AEC	Site not yet announced	Pressurized water	16.5	
AEC—Mobile Low Power Plant No. 1 (ML-1)	NRTS, Idaho	Gas cooled	0.4	

Source: Atomic Industrial Forum, Inc.

pated research and development results, construction could begin by 1965 on a plant of the boiling water type with internal nuclear superheating of the steam, which could reasonably be expected to produce power at a cost of 6.75 mills per kilowatt-hour. Expected results for several other reactor types range near these figures. It is estimated that nuclear power would begin to become meaningfully competitive at a cost in the range of 7 to 8 mills. A large nuclear power plant normally requires three to four years to construct.

United States Power Projects Summary. The reactor tables presented with this report summarize the latest basic information on new completions, construction starts, contracts and announced plans for United States nuclear power reactors. The overall status of United States nuclear power projects as of Sept. 1, 1959 is summarized in the "Progress Chart" accompanying this report.

Power Highlights. The Forum also noted progress at Shippingport, Pa., where the nation's first nuclear power plant began operating in 1957. This 60 emw. pressurized water plant, built by Westinghouse Electric Corp. and operated by Duquesne Light Co. was shut down in October for its first complete refueling operation, having generated over 388 million kw.-hr. of electricity. Duquesne reported that the failures of only one or two natural uranuim oxide fuel elements out of 95,000 such elements in the reactor is lower by a factor of hundreds than the design rate. The company compared cost of $14 million for two partial reloadings of the first core to an estimated cost of $16 million for the second core now being installed; improvements in the latter, resulting from design improvements suggested by operating experience, are expected to multiply perhaps five-fold the kilowatt-hour yield.

During 1959 an 110 emw. pressurized water reactor plant being built by Westinghouse for the Yankee Atomic Electric Co. at Rowe, Mass., neared 90% of completion of external building construction as reactor fabrication continued. Power Reactor Development Co. continued construction and began system testing of the 100 emw. fast breeder reactor plant which it will operate at Lagoona Beach, Mich., to generate electricity for The Detroit Edison Co. The pressurized water plant which Babcock & Wilcox Co. is building at Indian Point near New York City for Consolidated Edison Co. ended the year with 55% of the construction complete. The reactor will produce 155 emw.; a conventional superheater will add 104 emw.

Nuclear plants now building, in which the government is a partner, include Allis-Chalmers Manufacturing Co.'s 22 emw. boiling water plant (including a conventional superheater) for Rural Cooperative Power Association in Elk River, Minn., and Atomics International's 75 emw. sodium graphite plant for Consumers Public Power District at Hallam, Nebr. Ground was broken during the year for AI's 11.4 emw. organic moderated plant for the city of Piqua, Ohio.

Two large gas cooled reactors have been announced, one involving a guarantee by General Dynamics Corp. that it will build a reactor at a fixed price and return $14.5 million for research and development if the reactor is not operated as part of an established power system. GD and Philadelphia Electric Co. in August signed a contract covering AEC assistance for a privately owned, 30 to 40 emw. advanced high temperature gas cooled reactor (HTGCR) for which ground will soon be broken at Peach Bottom, Pa.

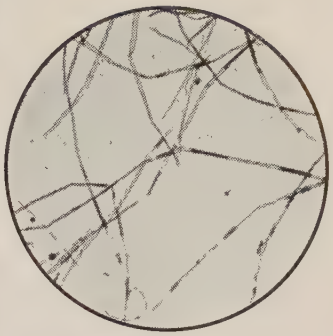
The cost of the project, estimated at $44.2 million, will be divided among: the AEC ($14.5 million toward research and development); High Temperature Reactor Development Associates, Inc., a nonprofit association of 52 electric utility companies including Philadelphia Electric ($16.5 million toward construction); and Philadelphia Electric ($8 million more toward construction). Philadelphia Electric will own and operate the plant as part of its power system and will also pay for land, interest, and fabrication of the first two fuel cores.

The $14.5 million which General Dynamics will receive from AEC under the third round of the Power Demonstration Reactor Program is conditional upon the completion of the project as an operating plant, estimated for 1963. The AEC will waive all or most fuel use charges during the first five years of operation.

Ground was broken in 1959 for the second large gas cooled reactor, the AEC owned, $30 million, 25 emw. experimental gas cooled reactor (EGCR) designed both as a prototype demonstrating currently available technology, and as a flexible testing facility. Principal design is being performed by Kaiser Engineers and Allis-Chalmers Manufacturing Co., with Oak Ridge National Laboratory designing fuel elements and control rods and Tennessee Valley Authority the primary power lines and substation. Kaiser is architect-engineer for the plant which TVA will operate near Oak Ridge, Tenn., with completion scheduled late in 1962.

Power Sales Overseas. In 1959 Westinghouse signed a contract with Socieàt Elettronucleare Italiana (SELNI) of Italy, for a 165 emw. pressurized water nuclear plant to be built near Milan. General Electric began negotiations with the Japan Atomic Energy Research Institute for a 12.5 emw. boiling water reactor to be built at Tokai-Mura, Japan; and with Suisatom for a 15 emw. boiling reactor for a plant at Villigen, Switzerland.

American hopes for an expanded overseas market for nuclear power equipment as a result of the Euratom program, however, received relatively little encouragement during 1959, the Forum review indicates. Utility organizations within the six Euratom nations of western Europe were invited this year to submit proposals for construction of nuclear power plants of United States design (and of combined United States and European manufacture) by 1963. The


Celgar Kraft will contain long, thick-walled fibres

1. High Tearing Resistance

Celgar Kraft will contain long, strong fibres such as these from Interior-grown western hemlock. They will give the final paper product the high tearing resistance which is the outstanding characteristic of Western Canadian kraft pulps.

Celgar Kraft

V5763-1

United States-Euratom Joint Reactor Board, in Brussels, has reported that each of five utility groups has reaffirmed its intention expressed earlier in the year to participate in the United States-Euratom program. However, only one out of the five "fully met the requirements" of the invitation. This organization is Italy's Società Elettronucleare Nazionale (SENN), which plans to build a 150 emw. boiling water reactor at Punta Fiume between Rome and Naples. The reactor is to be supplied by General Electric Co., and the plant will be financed in part by the World Bank under arrangements initiated more than two years ago. The response from the remaining four utility groups can be described as follows:

Arbeitsgemeinschaft Baden-Württemberg zum Studium der Errichtung eines Kerkraftwerkes (AKS) of Germany proposes to build an organic moderated reactor of 150 emw. electrical capacity. The plant is to be located at a site as yet unspecified in Baden-Württemberg. Prime contractor for the nuclear portion of the plant is Atomics International, which will work with its German affiliate, Interatom. Brown Boveri will act as architect engineers and prime contractor for the conventional portion of the plant. The German federal government has expressed its readiness to cover part of the difference between conventional and nuclear costs up to 100 million German marks ($23.8 million).

Electricité de France, EDF, together with a Belgian company, Centre et Sud, plans to submit a proposal for a 150 mw. plant of yet unspecified design to be located in France at Chooz, near the Franco-Belgian border on the Meuse river. The site has already been surveyed, but definite proposals have not yet been completely elaborated.

The SEP, which groups all the producers of electricity of the Netherlands, has also expressed continued interest.

The West Berlin utility, BEWAG, intends to build a 150 mw. reactor.

In addition to development of nuclear reactors as sources of central station power, at home and abroad, the AIF reported 1959 progress in the use of reactors to propel not only merchant ships and naval vessels, but eventually, space rockets.

Savannah Launched. In July the *N. S. Savannah*, the world's first nuclear powered commercial cargo or passenger-carrying vessel, was launched at the Camden, N. J., yards of its builder, the New York Shipbuilding Corp. The Babcock & Wilcox Co. is manufacturing the ship's 70 tmw. pressurized water reactor. Total cost to the AEC and the Maritime Administration, who jointly sponsored the project, will come to more than $40 million. The *Savannah* is 595 ft. in length, will displace 22,000 tons fully loaded, and normal shaft horsepower is 20,000; normal speed is 21 knots. Present plans are for initial tests and trials extending from six months to a year. The States Marine Lines has been named operator of the vessel on its completion.

Nuclear Navy. The size of the nuclear Navy program has involved many United States manufacturers as principal contractors, and many more as subcontractors. Westinghouse is supplying reactors for 37 vessels, including the cruiser *Long Beach* and the aircraft carrier *Enterprise*. General Dynamics' Electric Boat Division had contracted for the construction of 11 submarines. Also supplying reactors to the nuclear Navy are General Electric and Combustion Engineering, and private shipbuilders include Ingalls, Newport News, New York Ship, and Bethlehem.

The year 1959 began with four submarines in active service. These were the *Nautilus*, the *Skate*, the *Swordfish*, and the *Sargo*. A fifth, the *Seawolf*, was temporarily out of commission while its sodium cooled reactor was replaced by a pressurized water reactor, to make for uniform servicing along with the rest of the nuclear Navy. The *Seawolf* is scheduled to return to operation about June, 1960.

During the course of the year five additional submarines came into operation: the fast attack submarine *Skipjack;*

Nuclear Test and Research Reactors

With a power level of 100 thermal kw. and above

Owner, designation	*Location*	*Type*	*Output, thermal kw.*	*Principal contractor*
Placed in operation in 1959				
Westinghouse Electric Corp.	Waltz Mill, Pa.	Tank	20,000	Westinghouse Electric
Industrial Reactor Laboratories, Inc.	Plainsboro, N. J.	Pool	5,000	American Machine & Foundry Co.
AEC—Brookhaven Medical Reactor	Upton, N. Y.	Tank	1,000	Brookhaven Natl. Laboratory
AEC—Shield Test Pool Facility (Susie)	NRTS, Idaho	Pool	100	General Electric Co.
AEC—Transient Reactor Test (TREAT)	NRTS, Idaho	Graphite	100	Argonne National Laboratory
Placed in operation abroad by U. S. manufacturers in 1959				
Comitato Nazional per le Ricerche Nucleare (CNRN)	Ispra, Italy	Tank	5,000	Allis-Chalmers Mfg. Co.
McMaster University	Hamilton, Ont.	Pool	1,000	American Machine & Foundry Co.
Societa Ricerche Impianti Nucleari (SORIN)	Saluggia, Italy	Pool	1,000	Allis-Chalmers Mfg. Co.
General Dynamics Corp.	U. S. Exhibit, World Agric. Fair, New Delhi, India	Pool	100	General Dynamics
Began construction or contracted for in 1959				
University of Buffalo	Buffalo, N. Y.	Pool	1,000	American Machine & Foundry Co.
Argonne Natl. Laboratory—(Juggernaut)	Lemont, Ill.	Graphite/water	250	Argonne National Laboratory
University of Illinois	Urbana, Ill.	Pool	100	General Dynamics Corp.
Texas A. & M. College	College Station, Tex.	Pool	100	General Dynamics Corp.
State College of Washington	Pullman, Wash.	Pool	100	General Electric Co.
Began construction or contracted for abroad by U. S. manufacturers in 1959				
Centro Autonomo Militare Energia Nucleare (CAMEN)	between Livorno and Pisa, Italy	Pool	5,000	Babcock & Wilcox Co.
University of the Philippines	Quezon City, The Philippines	Pool	1,000	General Electric Co.
Turkish Atomic Energy Commission	Cekmece, Turkey	Pool	1,000	American Machine & Foundry Co.
Natl. Atomic Energy Research Institute	Paktai-ri, Korea	Pool	100	General Dynamics Corp.
Plans announced in 1959				
AEC—Nuclear Test Plant (NTP)	NRTS, Idaho	Pressurized water	60,000	

the twin-reactor radar-picket submarine *Triton*, largest and most powerful submarine ever constructed; the fast attack sub *Seadragon*; the guided missile sub *Halibut;* and the first of the ballistic missile subs, the *George Washington*. These five vessels will bring the commissioned strength of the nuclear Navy to 10. Eleven additional submarines are scheduled for completion in 1960.

Notable nuclear Navy developments in 1959 also included the completion of the SIC land based prototype PWR for small submarine propulsion, built by Combustion Engineering, Inc. The first propulsion unit of this type is to be installed in the hunter-killer sub, *Tullibee*, now under construction by General Dynamics Corp. at Groton, Conn. In addition, the second reactor of the Large Ship Reactor Prototype (AIW) at the National Reactor Testing Station in Idaho went critical. This twin-reactor prototype was designed and developed by Westinghouse as a model for the cruiser *Long Beach* and the aircraft carrier *Enterprise*.

Keels were laid during 1959 for the submarines *Scamp, Permit, Plunger, Pollack, Barb, Tinosa*, and *Ethan Allen*, and for the destroyer leader *Bainbridge*. Launched during the year were the *Halbiut, Scorpion, Sculpin, George Washington, Patrick Henry, Theodore Roosevelt, Robert E. Lee*, and the cruiser *Long Beach*.

The authorized or existing nuclear Navy at the end of 1959 consisted of 43 vessels, powered by 53 reactors: 40 submarines each powered by a single reactor, except for the *Triton*, which has two reactors; one cruiser (two reactors); one destroyer leader (two reactors); and one aircraft carrier (8 reactors). Long lead items only have been authorized for three Polaris submarines and for a second aircraft carrier, the future of which remains in doubt.

Nuclear Rockets. In June Los Alamos Scientific Laboratory placed in operation at Jackass Flats, Nev., the Kiwi-A ground based experimental reactor as part of the Project Rover nuclear powered space rocket program. The reactor is mounted on a flatcar for ease of transfer from the testing site to assembly and disassembly shops, and is cooled with hydrogen gas which, in an actual rocket, would serve as the propellant. Kiwi-A, the first reactor experiment in the Rover program, is expected to be followed by other experimental reactors known as Kiwi-B, Dumbo, and Condor, the latter two possibly capable of flight.

Air Force contractors on the Rover Project for the development of materials and non-nuclear components and on other non-nuclear aspects include North American Aviation, Aerojet-General, ACF industries and Edgerton, Germeshausen & Grier.

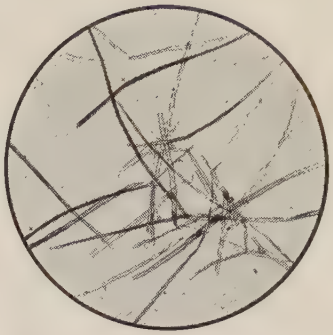
Power in Space. Near year-end Atomics International successfully developed and tested at Santa Susana, Calif., the high-temperature prototype reactor for the 3-kw. SNAP-II power source for space vehicle use. The complete device was described as weighing 220 lb. without shielding and being about the size of a milk can. The reactor is cooled with liquid sodium, which gives up its heat to mercury in a heat exchanger, with mercury vapor driving a miniature turbine to generate power. Fuel is enriched uranium. The power conversion system was developed by Thompson-Ramo-Woodridge, Inc., and has also been successfully tested using an electrical heat source.

The development cost for the device was about $6.5 million, and the unit cost for production models is expected to be $400,000. The device would be capable of supplying continuous power for at least a year, replacing the equivalent of "several hundred thousand pounds" of conventional chemical batteries. It could be ready for flight within a year. Other devices under the SNAP (Systems for Nuclear Auxiliary Power) program utilize the heat generated by radioisotopes which is converted directly to electricity through the use of thermocouples. Martin Co. is developing the SNAP-I and SNAP-III devices of this type.

Aircraft Nuclear Propulsion. Development continued during the year on two reactor concepts for aircraft propulsion. One of these is the direct cycle concept, being developed by General Electric Co., which had progressed to the stage of final design and initial manufacture for a ground test prototype for an actual airborne power plant when it was reoriented in October to the use of more advanced fuel elements giving higher performance than originally planned. The other approach is the indirect liquid-metal-cooled cycle, being pursued by Pratt & Whitney Division of United Aircraft Corp., which is still in the research and development stage with work on a reactor experiment now starting.

Process Heat. During the year the AEC and the Department of the Interior signed an agreement providing for the construction at Point Loma, San Diego, Calif., of a 40,000 thermal kilowatt process heat reactor of the pressurized water type, to be coupled to a million-gallon-per-day plant for the conversion of salt water to fresh water. Completion is scheduled for July, 1962, at an estimated cost of $4 million for the reactor and $2 million for the conversion plant. Sargent & Lundy is providing architect-engineering services for the reactor.

Thermal Breeder Program. In1 959 the AEC launched a centralized program aimed specifically at utilizing the energy latent in thorium to "breed" more nuclear fuel than is consumed in reactor operations. The AEC in its August announcement used the word "reorient" to describe consolidation of research on three fluid fuel reactor concepts into a single one to be selected next year. The concepts were aqueous homogeneous and molten salt at Oak Ridge, now headquarters for the new breeding program, and liquid metal fuel, now being terminated at Brookhaven National Laboratory, Upton, N. Y., and at the Lynchburg, Va., facility of the Babcock & Wilcox Co. Each of the three concepts had been launched with the hope of eventually achieving breeding,



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also will contain
thinner-walled fibres

2. High Bursting Resistance

Celgar Kraft also will contain thinner-walled fibres from such species as spruce, balsam and cedar. These impart to the final paper product the high bursting resistance which is the outstanding characteristic of Eastern Canadian and Scandinavian bleached kraft softwood pulps.

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but breeding development work or real scope had been postponed pending solution of basic operating problems. The new program is the first to concentrate on breeding as a specific goal: the development of a reactor which might convert enough thorium to U-233 in 25 years, beyond its own refueling needs, to provide an initial fuel charge for a second similar reactor. Funds were cut back from $19.7 million in fiscal 1959 to $7.5 for '60.

Food Irradiation. Plans have been deferred for an Army Ionizing Radiation Center at Stockton, Calif., for the study of high dose radiation sterlization of food. The project began in 1954 with an Army Quartermaster Corps announcement of a 5-yr. program for the study of food irradiation. Plans for the construction of a food irradiation reactor (by Kaiser Engineers) at the Sharpe General Depot at Stockton were abandoned in 1958 and an invitation was issued instead for the construction of a high intensity food irradiator (HI-FI) using a 2-million cobalt-60 source.

Approximately $1.3 million has already been spent on the overall project, which would have cost a total of $7.5 million. Curtiss-Wright Corp. had been awarded a $1.6 million contract by AEC for construction of the cobalt-60 HI-FI facility. The Army said that about $350,000 of this amount has been spent.

Industrial Uses of Radioisotopes. An AEC-sponsored survey by the National Industrial Conference Board produced, among others, the following points:

The total annual net savings "realized by major industries" from using radioisotopes amount to $39 million which is approximately one-tenth of AEC's 1957 savings estimate.

Petroleum refining and related industries report savings of more than $11.5 million, which in nearly twice the next largest savings category for a single industry (chemical and allied products), and represents 30% of the grand total.

The industries following next in order, with each realizing more than $2 million in annual savings, are tobacco manufacturers, rubber and miscellaneous products, paper and allied products, and engineering and architectural services. Also following, with the last industry on the list accounting for only $10,751 in annual savings, are transportation; primary metal industries; electrical machinery; machinery other than electrical; stone, clay, and glass products; crude petroleum and natural gas; food and kindred products; fabricated metal; textile mill products; metal mining; and electric and gas companies. By type of application, radioisotope gaging accounts for savings of $17.6 million which is 45% of the grand total. Research, manufacturing and processing, and radiography follow next in order as the applications accounting for the balance of total savings.

Both the AEC and NICB acknowledged the difficulty in attempting to assess the extent of isotope savings. As to what constitutes a savings, for example, NICB said: "In general, a savings was defined as a measurable difference in costs between the method used prior to the introduction of radioisotopes and the new radioisotope method."

The report also points out: "The total economic value of radioisotopes in industry cannot be accurately assessed. A product which is materially improved may not only return a higher profit to the maker, but may contribute lower costs and higher value to those who use it or resell it. . . . Although the returns on investment are very high in many instances, only a very small percentage of American industrial companies are availing themselves of the opportunities for cost savings and profit increases that radioisotopes offer."

Uranium. By the end of the year, according to the Forum, there were 24 uranium ore processing mills operating in the United States. As a result of their output plus overseas purchase commitments, the United States in 1959 found itself procuring a sizable surplus of uranium concentrates over present needs. Estimates of known reserves of uranium ore in the United States reached 88.9 million tons as of July, 1959, compared to a figure of 82.5 million tons at the end of 1958. Domestic production of concentrates reached a point where output exceeded the total of foreign purchases.

As a result of the general current oversupply of uranium, the AEC in November announced that it would not exercise its options to purchase additional uranium concentrates from Canada after March 31, 1962. The AEC, however, negotiated an agreement with the Canadian government whereby deliveries under the present contracts may be stretched out beyond their present expiration dates in 1962–63 through Dec. 31, 1966, with partial advance payment. Total amount to be purchased and the price to be paid are not affected by the stretchout. (Canada at the same time announced that it had entered into a similar agreement with the United Kingdom.)

The total amount of uranium which the United States has contracted for with Canada totals approximately 40,000 tons of uranium oxide at a total cost of about $1 billion.

Training and Small Research Reactors

Power level below 100 thermal kw.

Owner	*Location*	*Type*	*Output, thermal kw.*	*Principal contractor*
Placed in operation in 1959				
American Radiator & Standard Sanitary Corp.	U. S. Exhibit, Intl. Trade Fair, Tokyo, Japan, May, 1959	Graphite/water	10	American-Standard
Iowa State College	Ames, Iowa	Graphite/water	10	American-Standard
University of Puerto Rico	Mayaguez, P. I.	Pool	10	Atomics International
Veterans Administration Hospital	Omaha, Nebr.	Tank	10	General Dynamics Corp.
AEC—Fast Source Reactor (FSR)	NRTS, Idaho	Fast	1	Argonne National Laboratory
West Virginia University	Morgantown, W. Va.	Pool	75 watts	Aerojet-General Nucleonics
University of Wyoming	Laramie, Wyo.	Homogeneous	10 watts	Atomics International
Placed in operations abroad by U. S. manufacturers in 1959				
Milan Polytechnic Institute	Milan, Italy	Homogeneous	50	Atomics International
Walter Reed Army Medical Center	Washington, D. C.	Homogeneous	50	Atomics International
University of California	Los Angeles, Calif.	Graphite/water	10	American Machine & Foundry Co.
University of Kansas	Lawrence, Kan.	Pool	10	Bendix Aviation Corp.
University of Maryland	College Park, Md.	Pool	10	Allis-Chalmers Mfg. Co.
North Carolina State College	Raleigh, N. C.	Pool	10	Nucledyne Corp.
Ohio State University	Columbus, Ohio	Pool	10	Lockheed Aircraft Corp.
Stanford University	Palo Alto, Calif.	Pool	10	General Electric Co.
Virginia Polytechnic Institute	Blacksburg, Va.	Graphite/water	10	American-Standard
University of Washington	Seattle, Wash.	Graphite/water	10	American Machine & Foundry Co.
Worcester Plytechnic Institute	Worcester, Mass.	Pool	1	General Electric Co.
Under contract by U. S. manufacturers abroad in 1959				
St. Paul's University	Yokosuka, Japan	Pool	Up to 100	General Dynamics Corp.

Canada is currently producing uranium oxide at the rate of approximately 15,000 tons per year, most of which is being purchased by the United States, with the United Kingdom a secondary market.

During the year the United States arranged to discontinue operation at the only government owned uranium milling facility, the 300-ton-per-day plant at Monticello, Utah, which was operated by the National Lead Co. Also during the year two privately owned and operated facilities came into production, the 492-ton per day Globe Mining Co. mill in Natrona County, Wyo. (as a subsidiary of Union Carbide Nuclear Co.), and the 522-ton per day mill of the Federal-Radorock-Gas Hills Partners near Riverton, Wyo. Construction of the latter mill began in April, 1959, and the first concentrates were produced in November.

U-235 Prices. At the end of the year, a three-man committee established by the Forum at the invitation of the AEC examined on a classified basis the AEC's cost and price structure for the production of uranium enriched in the U-235 isotope. The committee concluded that the present prices are without subsidy and may in fact be capable of future reduction, and that production capacity will be ample to support 40 million kilowatts of nuclear power capacity built over the next 15 years. The committee also concluded that new plant capacity for the production of uranium of low enrichment could be built without raising current U-235 costs.

Questions and Answers

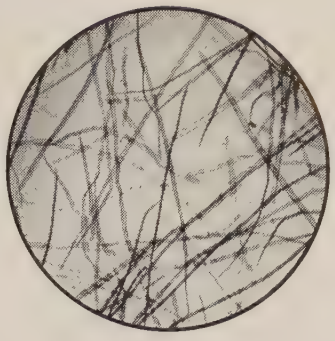
Good engineering questions are solicited as well as answers or alternate answers to the published questions. Material should be sent to TAPPI Headquarters, marked for the attention of H. O. Teeple. The items contributed will be forwarded to the Question and Answer Committee for processing. If comments are to be contributed, please refer to the question number.

* (1-10/60) *Question* (**Compound-Wound Motor Reverses When Starting**): **What would cause a compound-wound motor to reverse rotation upon reaching the third point of the starting rheostat? What would cause the same motor to run within normal speed limits at one starting and accelerate to a dangerous speed at another starting?**

* (*1.1-10/60*) *Answer:* The trouble is caused by the series and shuntfield windings being connected in opposition. The starting resistance was cut out at a rate that allowed the armature current to build up to a value, when the third point of the starter was reached that made the ampere-turns of the series winding greater than the shunt-winding ampere-turns. This reversed the polarity of the pole-pieces and consequently reversed the direction of the motor. In general, if a compound-wound motor with its field winding connected in opposition is starting under light load and is accelerated slowly, it will come up to normal speed without giving any evidence of anything being wrong. However, if an attempt is made to accelerate the same motor rapidly, it will reverse before the starting resistance is cut out, as explained in the foregoing, or it may run at a dangerous speed due to the demagnetizing effect of the series-field winding.

* (2-10/60) *Question* (**Carbon Brush Wear**): **In one of our d.c. motors that has four brushes on the commutator, two brushes wear away faster than the other two. What would cause this uneven wear when all four brushes are the same grade?**

The questions and answers in this section are reproduced courtesy *Power* magazine.



Celgar Kraft will have balanced properties

3. Balanced Properties

Through its mixture of long, strong, thick-walled fibres and its thinner-walled fibres, and due to the climatic conditions influencing tree growth, Celgar Kraft will achieve naturally a balance of strength properties which can normally only be duplicated by blending several grades of pulp.

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* (*2.1-10/60*) *Answer:* The brushes from which the current flows into the commutator always wear away faster than those through which the flow is from the commutator. The brushes through which the current flows into the commutator would be positive on a motor and negative on a generator. The additional wear on these brushes is due to small particles of carbon carried by the current from the brush contact surface and deposited on the commutator. If two of four brushes, of the same polarity, were to wear away faster than the other two, a difference in resistance in the shunts from the brushes to the brush-holder, or a difference in the tension on the brushes, would cause an unequal distribution of the current between the brushes and unequal wear.

* (3-10/60) *Question* (**Safety Valves for Superheaters**): **Should superheaters have safety valves attached?**

* (*3.1-10/60*) *Answer:* All superheaters should be equipped with safety vales, which should be set slightly lower than the boiler valves. With the superheater valves blowing first, a flow of steam through the superheater is assured should the load be suddenly thrown off the boiler, and there will be no danger of burning superheater tubes, as would be the case were the boiler valves to blow first. Again, the use of safety valves on the superheater outlets will prevent highly heated steam passing to the auxiliaries should the main power units be suddenly shut down.

* (4-10/60) *Question* (**Changing the Speed of a Fan Motor**): **Is it possible to increase the speed of direct-current motors used to drive fans? If so, how is it done?**

* (*4.1-10/60*) *Answer:* If a fan is driven by a series motor, the speed of the motor may be varied by connecting a resistance in parallel with the field winding. If the motor is of shunt type, its speed may be varied by connecting a resistance in series with the field winding. The value of the resistance will depend upon how much the speed is increased and upon the size of the motor. It is doubtful if a motor that has been designed to drive a fan of a given size can have its speed increased very much without being overloaded, since a fan load increases as the cube of the speed.

* (**5-10/60**) *Question* (**Fuse Blown on Three-Phase Motor**): **What would be the effects of a fuse blowing in either of the primaries or secondaries of three transformers connected in star, to a three-phase motor?**

* (*5.1-10/60*) *Answer:* The opening of a fuse on either side of the transformer bank would leave the motor operating single-phase. If the motor is lightly loaded it will continue to run until shut down, but cannot be started again until the fuse is replaced. While the motor continues to operate single phase it will take about 100% more current in the active phase than when operating three-phase. Single-phase pull-out torque will be from 30 to 40% of what it was for three-phase operation.

* (**6-10/60**) *Question* (**2200-V. Motor on 330 V.**): **What horsepower would a three-phase, 50-hp., 2200-v., 750-r.p.m., motor develop if connected on a 440-v. line?**

* (*6.1-10/60*) *Answer:* Theoretically, one fifth of 50, or 10 hp. and the starting torque would be $^1/_{25}$ of that at 2200 v. The only thing to do is to reconnect the stator winding for 440 v. Just what the grouping should be cannot be determined without knowing the number of coils and how they are grouped in the 2200-v. winding. If the 2200-v. winding is connected in series star, which is probably the case, then the 440-v. winding may be connected five-parallel star. However, if the number of coils per phase cannot be divided by 5 it may be somewhat of a problem to distribute the coils so as to get a satisfactory grouping. This gives an equivalent 418-v. winding, which is close enough to 440 v. to operate satisfactorily.

* (**7-10/60**) *Question* (**Induction-Motor Winding Bracing**): **Why are insulated rings placed over the ends of induction motor coils where they extend beyond the stator core, in some cases on both ends and in others on one end only?**

* (*7.1-10/60*) *Answer:* Frequently, where the coil throw is long or if the coils are not mechanically rigid, the ends are liable to be distorted when a heavy current flows through them. To prevent this, insulated rings are placed around the outside of the winding and the coil ends laced to these rings with heavy cord. This practice has been found to increase the life of the coils, since it tends to prevent any vibration of the coil ends that would otherwise occur. Where the throw of the coils is short the rings are only put on the end of the winding opposite to the one where the cross-connections are made, the cross-connection giving the necessary support to the coil ends on the end of the stator winding where the connections are made.

(**8-10/60**) *Question*: **An extremely old fourdrinier wet end shows abnormally high incidence of wire failures at the edges and seams. The edges are cracking frequently and closely, and there are whole patches of wire where the warp wires have broken. Edge relief, plastic edging, and changes in the type of wire have failed to show material improvement. What are possible causes for such failures, and probable cures?**

(*8.1-10/60*) *Answer:* Two sorts of bending may cause your trouble. Firstly, on an old machine roll diameters may be far below present practice, so that when a wire wraps around the roll it is subjected to high bending stress. With ordinary nonferrous wire materials this results in work hardening of the wire, which in turn contributes to bending fatigue failures of the warp wires. In addition, small diameter rolls may be sagging in the middle, which may relieve the tension at the center of the wire while maintaining it at the ends. This would result in over stressing of the edges and acceleration of failures there. Therefore if your failures are uniformly distributed across the wire, look merely for insufficient roll diameter, but if they show higher incidence toward the edges than in the center look for bending of a roll as well.

* (**9-10/60**) *Question* (**Induction - Motor Rotor Connections**): **I have rewound the rotors of a number of wound-rotor type induction motors and in each case the winding was connected star. Can the delta connection be used in grouping these windings? If so, why is it not used more generally?**

* (*9.1-10/60*) *Answer:* Either connection may be used, but in small and medium motors the star connection gives a more statisfactory winding design. The star connection gives a terminal voltage 73% higher at the slip rings than the delta connection. This is very desirable where the rotor is bar wound, since with this type of winding the voltage is comparatively low, and the current relatively high. With the star connection the current will be 58% of that obtained with the delta connection. Consequently, the starting resistance is much cheaper to construct. In some large-sized motors, however, the voltage produced in the rotor winding with a star connection may be so high as to be difficult to insulate and also require specially design central equipment to make them safe for an operator to handle. In such cases the rotor is connected delta, as this grouplng generally gives a voltage at the slip rings that is of a safe value.

* (**11-10/60**) *Question* (**Distance of Pully Centers for Motor Drive**): **What is the approximate rule for the minimum distance between pulley centers for open belt drives from small electric motors?**

* (*11.1-10/60*) *Answer:* A rule that has given satisfaction with pulleys of usual sizes is that the distance between the pulley centers should not be less than three times the sum of the diameters of the pulleys. A distance of four or five times the sum of diameters will result in a better drive.

* (**12-10/60**) *Question* (**Effect of Power Factor on Single-Phase Wattmeters**). **When two single-phase wattmeters are measuring power and power factor on a three-phase circuit, are meter readings adversely affected if power is received at a low power factor?**

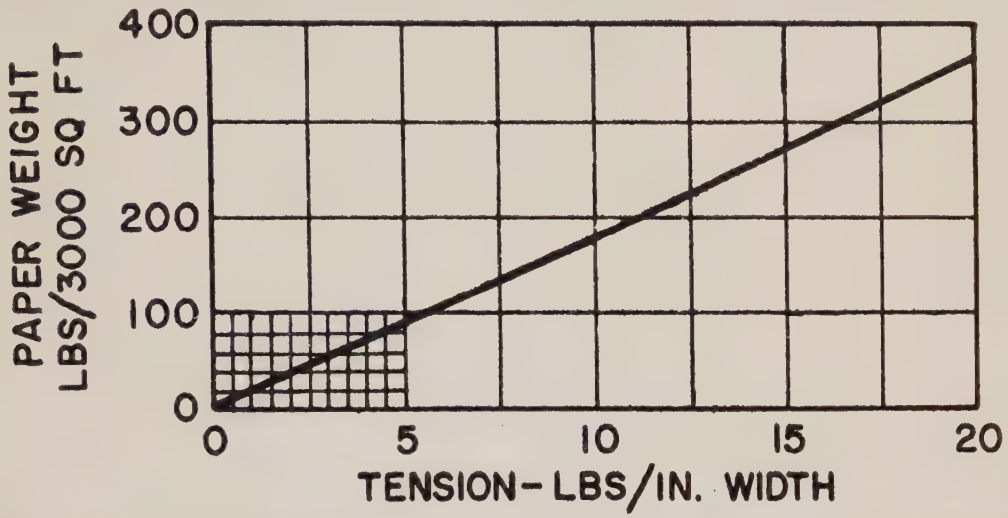
* (*12.1-10/60*) *Answer:* Power factor of a medium-voltage line is determined by loads connected to it, not by any condition in the line. Where three-phase power is measured by two single-phase meter at power factor under 100%, the reading of one meter will increase and that of the other decrease until at 50% power factor, one meter will reverse, and total power supplied is then the difference in the readings.

Power Requirements for Paper Machines

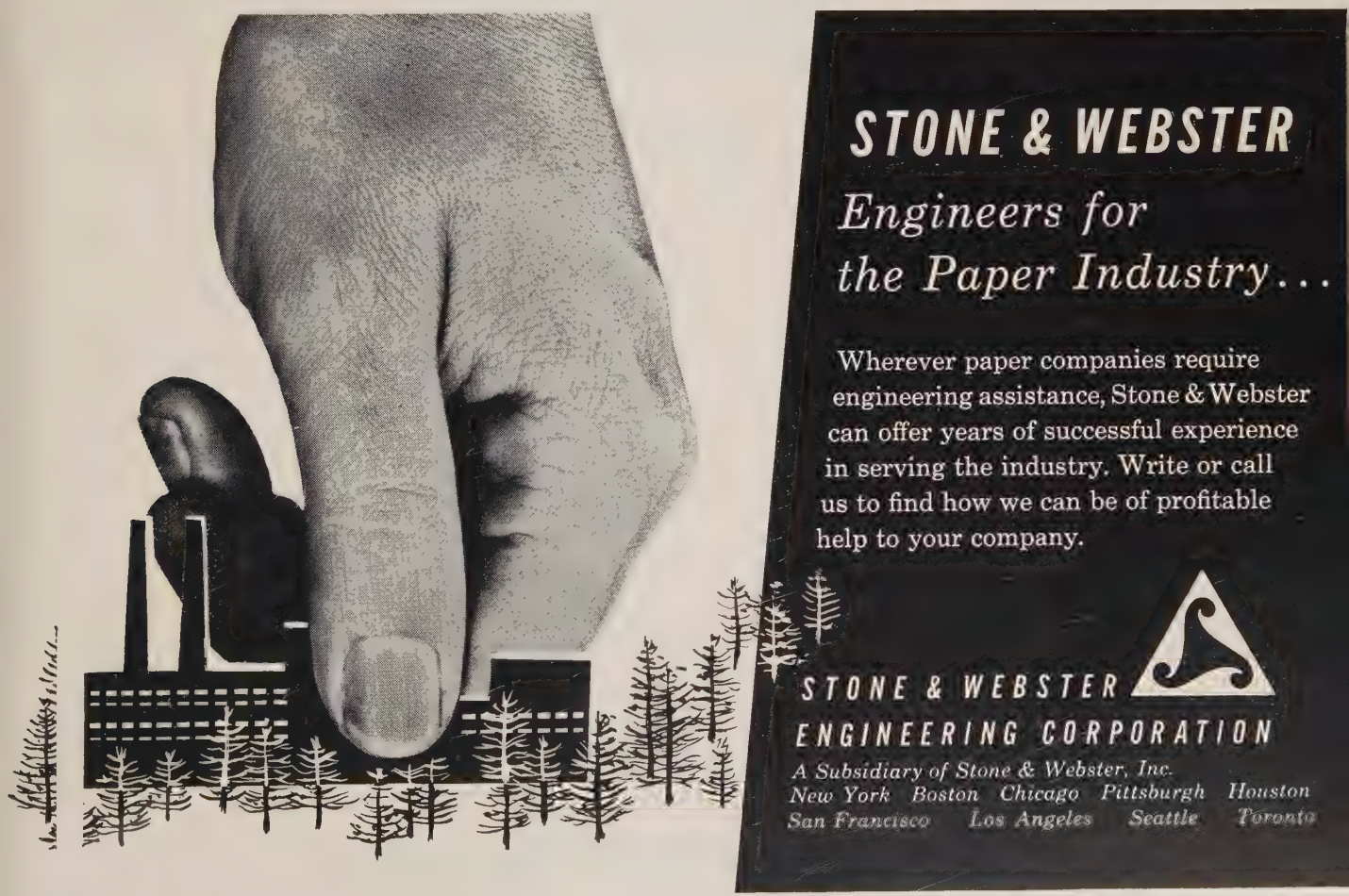
Paper Tension Versus Ream Weight Mean of Experience Values

TAPPI Data Sheet 206

(THIS DATA SHEET UNDER THE JURISDICATION OF THE ELECTRICAL ENGINEERING COMMITTEE)



Note: For examples in the use of Data Sheets Nos. 206, 207, and 208 see report on winder drive power requirements.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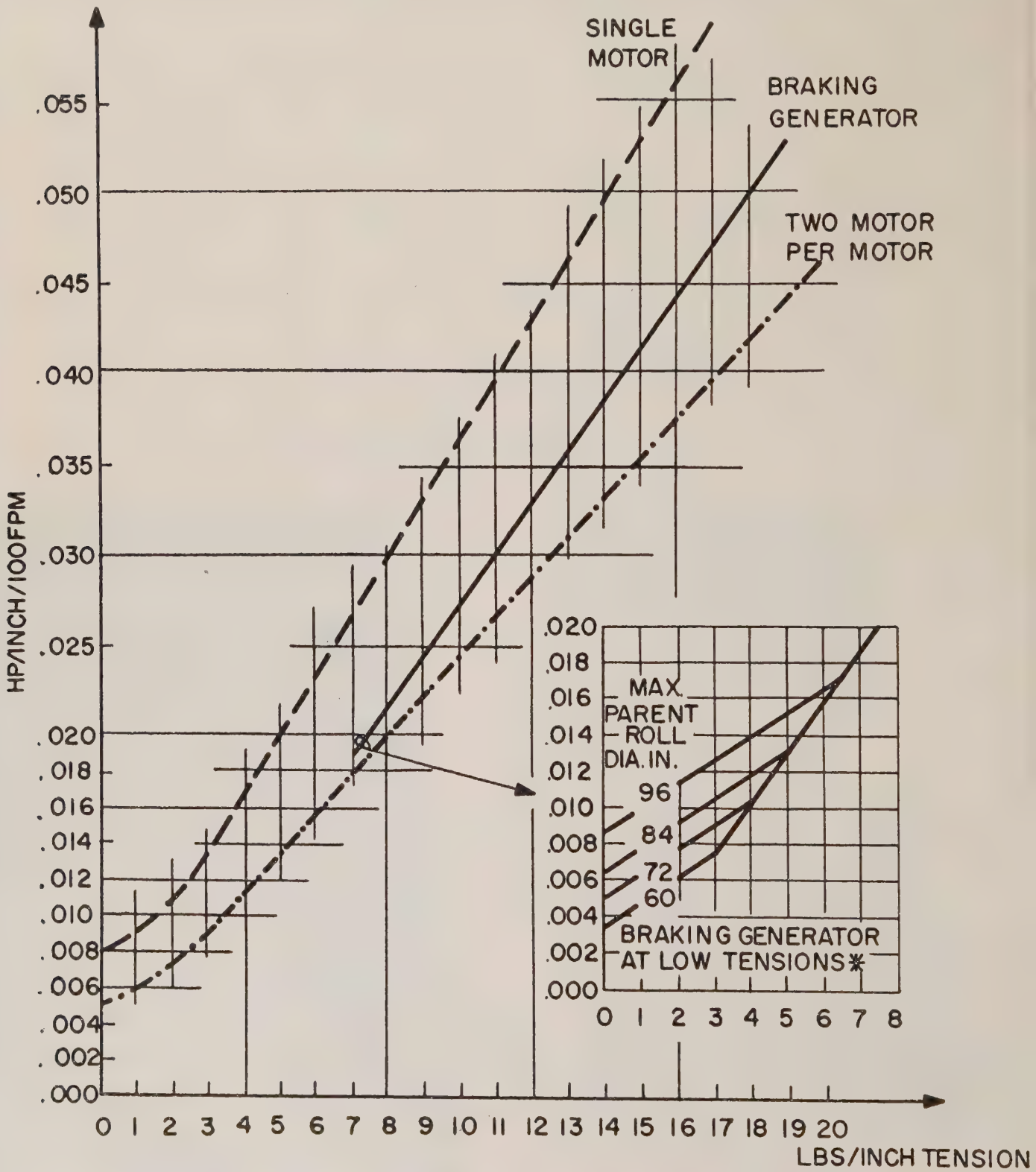
Power Requirements for Paper Machines

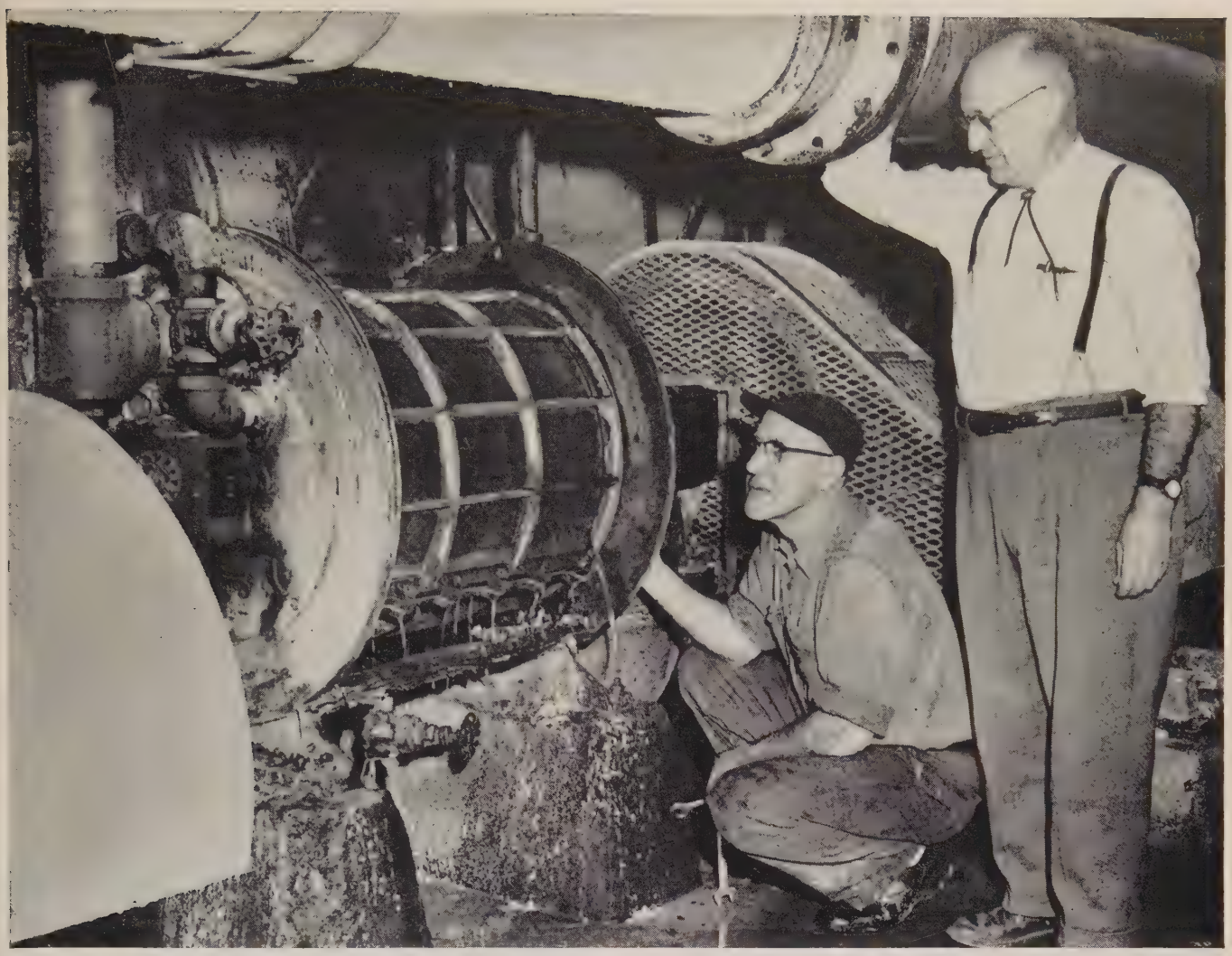
Winder Drive Power (hp.) Requirements

TAPPI Data Sheet 207

(THIS DATA SHEET IS UNDER THE JURISDICTION OF THE ELECTRICAL ENGINEERING COMMITTEE)



* Based on 120 $\frac{\text{f.p.m.}}{\text{sec.}}$ decel. rate with sheet intact and 200% rated load. Paper strength may limit acceleration rate on mechanical backstand winders.



St. Regis takes off the wraps to show you how they save $4000 yearly on screen plates

Until 1955, St. Regis Paper Company replaced all double plates on its fifteen rotary groundwood pulp screens four times a year. One hundred and twenty plates per year!

Since 1955, St. Regis has replaced only twelve of these plates per year, 1/10 as many. The saving? Approximately $4000 per year, by now a tidy $20,000.

How do they do it?

Two St. Regis men took the cover off one of the rotaries. You can see the plates. They are Inconel* nickel-chromium alloy.

If you were there, you would notice two things at once. First, you would see a remarkable absence of the metal loss that enlarges the slot areas and permits fibre to slip through in "bundles." (And eventually, of course, leads to excessive breakage.) Second, your fingertips would soon disclose that edges are smooth. Nothing to start build-ups of fibre strings.

Now here is a curious coincidence. In a totally independent 52-day, in-plant corrosion test of proposed rotary screen materials processing groundwood stock, Inconel alloy corroded just exactly 1/10 as much as phosphor bronze.

Experience shows, too, that applied stress, crevices, welding, intermittent wetting, and/or vapor phase exposure do not promote stress-corrosion cracking, intergranular corrosion or other selective attack on Inconel alloy. Not even if the medium includes chlorides and oxygen.

Are Inconel alloy rotary screen plates readily available?

Getting Inconel alloy screen plates is no problem. The metal itself is a warehouse item. And any number of fabricators know exactly how to bring out the best in the alloy and some make Inconel alloy screen plates regularly. We'll be glad to give you names. Maybe, too, there are other ways we can help you improve metal performance. We'll gladly try. Write:

*Inco trademark

HUNTINGTON ALLOY PRODUCTS DIVISION
The International Nickel Company, Inc.
Huntington 17, West Virginia

INCONEL®

RC-221. Fiber Identification (Various Stains) (Correction)

The following is an outline of the methods used in a large industrial laboratory for identifying the more common papermaking fibers. The various stains and their application have been designed in each instance to give the maximum color differentiation possible so that anyone reasonably familiar with the general technique may identify the fibers of an unknown sample.

Many of the stains given in the literature are of value only to those who are constantly making fiber analyses because clear differentiation is difficult for the untrained eye of the papermill chemist or technical man who is called upon only occasionally to identify an unknown paper.

The five stains to be described and their methods of use will provide a clear differentiation between all the common papermaking fibers in all possible combinations. They have been thoroughly tested on many fibers, for example, bleached, semibleached, and unbleached kraft pulps from many sources including Scandinavian, western, midwestern, eastern, southern, and Canadian krafts.

Component Solutions for the Stains

No. 1. Regular Herzberg iodine:

100 ml. distilled water
22 g. KI
2 g. crystalline iodine

Keep in a black or taped bottle for about 9 months.

No. 2. Modified Herzberg iodine:

100 ml. distilled water
6 g. KI
1.5 g. crystalline iodine

Keep in a black or taped bottle. Stable for about 9 months.

No. 3. Chloride:

50 g. zinc chloride
15 g. anhydrous calcium chloride

Makeup to a volume of 100 ml. with distilled water. Stable about 9 months.

No. 4. Salt:

Saturated sodium chloride solution.

No. 5. Potassium ferricyanide:

5 g. potassium ferricyanide, $K\ Fe_3(CN)_6$
50 ml. distilled water
50 ml. ethyl alcohol

No. 6. Ferric chloride:

5 g. ferric chloride, $FeCl_3$, 6 H_2O
100 ml. distilled water

No. 7. Bordeaux:

5 g. Du Pont Pontamine Bordeaux B
100 ml. distilled water

No. 8. W-basic orange:

2 g. basic orange
50 ml. distilled water
50 ml. ethyl alcohol

Note: Basic orange sold under any of the following trade names may be used:

Euchrysine 3RXA (General Dyestuff Corp., New York, N. Y.)
Phosphine 2RN (National Aniline Division, Allied Chemical Corp., New York, N. Y.)
Acridene Orange RS or LPS (Canadian Industries, Ltd., Montreal, Que.)

No. 9. Basic green:

0.75 g. Du Pont brilliant green crystals
25.5 ml. ethyl alcohol
11.0 ml. distilled water
62.5 ml. W-basic orange stain no. 8

No. 10. Brilliant green:

1.5 g. Du Pont brilliant green crystals
70 ml. ethyl alcohol
30 ml. distilled water

No. 11. Y-basic orange:

20 ml. distilled water
40 ml. ethyl alcohol
40 ml. W-basic orange stain no. 8

No. 12. Salt-glycerine:

50 ml. saturated sodium chloride (solution No. 4)
50 ml. glycerine

These 12 solutions should be kept in small bottles equipped with medicine droppers having uniformly sized tips. It is well to mark on each label the date the solution was prepared.

Slide cleaning solution:

50 ml. ethyl alcohol
50 ml. water
1 ml. concd. HCl (37%)

This solution will be found very effective for final cleaning of slides and cover glasses.

Preparation of Slides

Rule off one inch from each end of a clean slide with a glass-marking pencil. Place the slide over a low temperature hot plate (50 to 60°C.). Defiber the sample in the usual manner and make a fiber suspension of about 0.05 to 0.08%.

Place about 0.5 ml. of the fiber suspension on each end of the ruled slide. The ruled lines will keep the fibers within the marked area. As the water evaporates, tap the slide gently and arrange the fibers with a needle.

Note: The hot-plate temperature should be sufficiently low so that no, or at least very few, bubbles form as the slide dries. If, unavoidably, the surface temperature of the hot plate is too high, it will then help to place two half-inch wide metal strips on the surface of the hot plate and lay the slides on these strips.

RC-286. Analysis of Salt Cake (Colorimetric Determination of Magnesium)

In the TAPPI Standard T 619 m-53 magnesium is determined gravimetrically. However, magnesium can be determined more rapidly by a colorimetric method. The method is as follows:

Pipet 20 ml. of solution A (equivalent to 1.0 g. of sample) into a 100 ml. volumetric flask. Add the following solutions: 0.5 ml. of 1% H_2SO_4, 20 ml. saturated $CaSO_4$, and 5.0 ml. alum solution ($Al_2(SO_4)_3 \cdot 18H_2O$, 0.31 g.p.l.). Bring the volume to about 80 ml. with distilled water and mix. Add 5.0 ml. of 1% Methocel solution, 2 ml. of 0.05% Brilliant Yellow solution, and 4.0 ml. of 20% NaOH. Dilute to the mark; shake vigorously; wait 5 to 10 min. for full color development.

Read the color on a suitable spectrophotometer which has

been previously calibrated with prepared solutions of known magnesium. All readings should be at wavelength 520 mμ.

Submitted by

Richard J. Burrock
Minnesota & Ontario Paper Co.
International Falls, Minn.

RC-287. Analysis of Weak Black Liquor for Active Alkali

Knowledge of the amount of active alkali in weak black liquor may be useful in kraft mill chemical recovery calculations. The active alkali is determined in a manner similar to that often used for white liquor. Owing to the formation of a bulky precipitate of barium salts of organic acids, it is best to dilute to a large volume and use a large portion of that for testing.

To 50 ml. of weak black liquor in a 500 ml. volumetric flask, add 50 ml. of a 20% solution of barium chloride. Fill the flask to the mark with distilled water, shake well and then allow to stand until there is sufficient clear liquid to pipet 50 ml. for titration. Pipet the 50 ml. into a porcelain dish and dilute with 200 to 300 ml. of distilled water. Add 5 ml. of formaldehyde to insure complete titration of sulfides. Titrate with 1*N* HCL using Fleischer methyl purple indicator. The color change can be seen against the white porcelain at the edge of the liquid. In the calculations 1 ml. of 1 *N* HCl equals 0.387 lb. Na_2O per cubic foot.

Submitted by

Richard J. Burrock
Minnesota & Ontario Paper Co.
International Falls, Minn.

Chemical Pulping Processes

Chemipulp and Chemipulp-KC systems held achieve maximum production of uniform, high quality pulp at lower cost and provide means for efficient recovery of heat and chemicals.

- Hot Acid Systems
- Jet-Type Sulphur Burners
- Independent Recovery Systems
- Spray-Type SO_2 Gas Cooling Systems
- Recovery Towers
- Sulphite Acid Systems
- Digester Circulating Systems
- Neutral Sulphite Semi-Chemical Liquor Plants
- Chip Distributors
- Hydroheaters
- Black Liquor Oxidation Systems for Sulphate Pulp Mills

Chemipulp Process Inc.
Watertown, N. Y.

Associated with
Chemipulp Process Ltd., 253 Ontario St., Kingston, Ont.
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Pacific Coast Representative
A. H. Lundberg Inc., P.O. Box 186, Mercer Island, Wash.

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LOCAL SECTION ACTIVITIES

Meeting Calendar

October, 1960

27 Delaware Valley Section, Engineers Club, Philadelphia, Pa. Panel on "Design in Packaging."

November, 1960

3 Lake Erie Section, Cleveland, Ohio.

4 Central District, Empire State Section University Club, Syracuse, N. Y. "Plastic Wires," by a representative of Formex Co.

9 Metropolitan District, Empire State Section, 465 Lexington Ave., New York, N. Y. "Investing in the Paper Industry," by Louis Stevenson, Consultant, Tucker, Anthony & R. L. Day.

10 Northern District, Empire State Section. Mill Trip to Crown Zellerbach Corp., Carthage N. Y. Dinner at Johnnies Inn, Deferiet, N. Y.

14 Eastern District, New England Section, Armand's Beacon Terrace, Framingham, Mass.

15 Ohio Section, Manchester Hotel, Middletown, Ohio. Joint meeting with PIMA "Significance of Election Results as They Pertain to Industry." Congressman Paul F. Schenck, Third Ohio District.

16 Pacific Section, Salem, Ore., "Modernization."

17 St. Louis Section, Le Chateau Restaurant, 10405 Clayton Rd., St. Louis County, Mo. "Recent Advances in the Packaging Field," by C. P. Olstad, Crown Zellerbach Corp.

18–19 Southeastern Section, Dinkler Plaza Hotel, Atlanta, Ga.

December, 1960

2 Central District, Empire State Section, University Club, Syracuse, N. Y. "Nip Impressions, by a representative of Stowe-Woodward Co.

5 New England Section, Storrowton Tavern, West Springfield, Mass.

8 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. Students' Night. Speaker: P. E. Nethercut.

9 Delaware Valley Section, Engineers Club, Philadelphia, Pa. "Stock Cleaning."

13 Metropolitan District, Empire State Section, 465 Lexington Ave., New York, N. Y. "Dyeing of Paper," by Joseph Gessner, Head of Paper Division, E. I. du Pont de Nemours & Co. Inc.

15 Ohio Section, Manchester Hotel, Middletown, Ohio. "Plastic Fourdrinier Wires," by Charles Lee, Formex Co.

January, 1961

6 Central District, Empire State Section, University Club, Syracuse, N. Y. "General Coating Methods," by George Booth, Black-Clawson Co

9 Eastern District, New England Section, Armand's Beacon Terrace, Framingham, Mass.

10 Metropolitan District, Empire State Section, 465 Lexington Ave., New York, N. Y. "State of the Industry," by W. R. Adams, President, St. Regis Paper Co.

10 Ohio Section, Manchester Hotel, Middletown, Ohio. "Some Recent Advances in Interfiber Bonding with Wet End Additives," by Merchant L. Cushing, A. E. Staley Mfg. Co.

12 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. "Problems in Research and Development for Small Specialty Mills," by F. C. Keeney.

17 Pacific Section, Tacoma, Wash., "Process and Quality Control Techniques."

20–21 Southeastern Section, Francis Marion Hotel, Charleston, S. C.

26 Delaware Valley Section, Engineer Club, Philadelphia Pa., "Packaging."

February, 1961

3 Central District, Empire State Section, University Club, Syracuse, N. Y. "Natural Resources of New York State as Related to the Pulp & Paper Industry," by Assemblyman R. W. Pomeroy and N. J. Stout.

9 Ohio Section, Manchester Hotel, Middletown, Ohio. "Trailing Blade Coating," by Harold Annis, Oxford Paper Co.
Northern District Empire State Section, Hotel Woodruff, Watertown.

March, 1961

2 Lake Erie Section, Cleveland, Ohio.

3 Central District, Empire State Section, University Club, Syracuse, N. Y. "New Developments in Corrugated Boxes," by Patrick Palmaccio, Mead Corp.

9 Northern District Empire State Section, Hotel Woodruff, Watertown.

13 Eastern District, New England Section, Armand's Beacon Terrace, Framingham, Mass.

14 Metropolitan District, Empire State Section, 465 Lexington Ave., New York, N. Y. "The Paper Industry in Europe," by W. W. Wilson, Editor, *Pulp & Paper*.

14 Ohio Section, University Center, Miami University, Oxford Ohio. "Appearance Properties of Paper," by Richard Hunter, Hunter Associates Laboratory.

21 Pacific Section, Longview, Wash., Shibley Award Meeting.

24–25 Southeastern Section, Bon Air Hotel, Augusta, Ga. Tour of Continental Can Co.

April, 1961

6 Lake Erie Section, Cleveland, Ohio.

7 Central District, Empire State Section, University Club, Syracuse, N. Y. Senior Night—Junior Awards.

10 New England Section, Storrowton Tavern, West Springfield, Mass. Management Night.

11 Metropolitan District, Empire State Section, 465 Lexington Ave., New York, N. Y. "The Reused Fiber."

13 Ohio Section. Tour National Cash Register Co. at Dayton, Ohio.

13 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. Joint meeting with Beaver Falls Chapter, A.I.C. "The Planning of a Technical Center," by K. A. Arnold, St. Regis Paper Co.

11 Northern District Empire State Section, Hotel Woodruff, Watertown. Annual Ladies' Night.
11 Ohio Section, Hartwell Recreation Hall, Cincinnati, Ohio. Ladies' Night.
19 Metropolitan District, Empire State Section, Tavern-on-the-Green, New York, N. Y. Ladies Night.
19–20 Southeastern Section, General Oglethrope Hotel, Savannah, Ga.

June, 1961

8–10 Empire State Section, Whiteface Inn, Whiteface, N. Y.
9–10 Joint Meeting, New England Section and Maine-New Hampshire Section, Wentworth-by-the-Sea, N. H.

June, 1962

1–2 New England Section, Chatham Bars Inn, Chatham, Mass.
7–9 Empire State Section, Whiteface Inn, Whiteface, N. Y.

June, 1963

6–8 Empire State Section, Whiteface Inn, Whiteface, N. Y.

June, 1964

11–13 Empire State Section, Whiteface Inn, Whiteface, N. Y.

EMPLOYMENT SERVICE

The rates per issue for Positions Open advertisements in *Tappi* are as follows:

Per line	$ 3
1/16-page Box	$ 50
1/8-page Box	$ 80
1/4-page Box	$125

Positions Wanted

E501-60. Chemical Engineer with long experience and wide acquaintance in pulp, paper, and paperboard and packaging on Pacific Coast seeks employment in that region with supplier to these industries. Desires sales and technical service work. Willing to travel extensively.

E502-60. Maintenance supervisor, 12 years experience in pulp and paper mill instrumentation and mechanical maintenance in Canadian newsprint, sulfite, groundwood mills desires position in the United States.

E504-60. Mature practical paper technician. Experienced in development and production of fine paper specialties. Available for technical, production, or coordinating job in progressive operation.

E505-60. Free lance agent with many top level contacts at paper mills in Great Britain, requires pulp agency. Substantial business could be available. Give full details.

E506-60. Paper School Graduate, 41 years old with 18 years experience including production management and supervision, production and new product development, technical director, quality control, and research and development in the manufacture of publication, sulfite and groundwood specialties, glassine, industrial specialties, rubber impregnated papers, waxing, and laminating. Pulp mill design and development.

Positions Open

P1021-60. Chemical Engineer for research and development work with growing specialty paperboard mill in Eastern New York State. Employees know of this ad.

ENGINEER

Graduate Engineer to reside in Sitka, Alaska as permanent member of engineering department. This person would be expected to assist with maintenance and design problems of an operating pulp mill located in southeastern Alaska. Write Personnel Supervisor, Alaska Lumber & Pulp Co., Inc., Box 1050, Sitka, Alaska.

(P1022-60)

QUALITY CONTROL SUPERVISOR

Excellent opportunity with well established photographic-sensitizing firm for man with B.S. in pulp and paper, forestry, or chemical engineering.

Will supervise Q. C. activities of raw stock material, including acceptance, testing, vendor liaison, production control, etc.

Kindly send résumé and salary requirements to:

Fred A. Weterrings
Industrial Relations Division
Haloid Xerox, Inc.
P.O. Box 1540 Rochester 3, New York

HALOID XEROX®

PROCESS SUPERVISOR

To be trained for Operating Supervisor involving Stock preparation and Processing Bagasse Insulation Board in Multi Board Machine Plant.

Prefer Technical, Chemical or Engineering background and some experience in paper, fiber board or related process industries.

Excellent career opportunity for rapid management progression in a growth company.

Write or Telephone:

J. L. Dickson
Works Manager
The Celotex Corporation
Marrero, Louisiana

(P1016-60)

CHEMISTS OR CHEMICAL ENGINEERS

An international Manufacturer of Paper and Paper Products has several openings for chemists or chemical engineers to work in the fields of product development and quality control. Salary is open. All replies held confidential. Please forward complete resume to P1023-60, **Tappi,** 360 Lexington Ave., New York 17, N. Y.

CHEMIST OR PROJECT ENGINEERS

Chemist, mechanical, electrical or chemical engineer wanted for expanding development program with modern gravure publication printing company. We want a technical man, well grounded in theory and with the courage of his own convictions. Recent graduate will be considered, but any experience in graphic arts, including paper manufacturing, converting, or allied industries is preferred. Salary open.

Contact: **Mr. D. A. Cutler**
Director of Research & Engineering
Triangle Publications, Inc.
440 North Broad Street
Philadelphia 1, Penna.

(P1024-60)

PROCESS ENGINEER

Graduate Chemical Engineer with experience in sulphite cooking and bleaching preferably of dissolving pulp. This pulp mill located in southeastern Alaska Write Personnel Supervisor, Alaska Lumber & Pulp Co., Inc., Box 1050, Sitka, Alaska.

(P1025-70)

RESEARCH AND DEVELOPMENT

Chemist, chemical engineer or paper school graduate, preferably with experience in paperboard coating. Excellent opportunity for advancement. Pleasant working conditions and many company benefits. Medium size mid-western integrated paperboard operation.

Reply to P1026-60, Tappi, 360 Lexington Ave., New York 17, N.Y

OPPORTUNITY IN ELECTRICAL INSULATING FIELD

New England specialty paper manufacturer has challenging staff position to fill in newly expanded laboratory facilities in the field of Electrical Papers.

Major field of emphasis will be in research, development, and process control for the manufacture of electrical insulating papers. Knowledge of dielectric media including paper, oils and resins, and experience with electrical testing procedures highly desirable. Applicant should be capable of carrying out independent investigations with a minimum of direction. Minimum rquirement for the above position is a BS degree. Experience in the paper industry and/or insulating field advantageous.

Please send resume of your experience, education and salary requirements to:

HAROLD W. KNUDSON, TECHNICAL DIRECTOR
HOLLINGSWORTH & VOSE COMPANY
EAST WALPOLE, MASS.

(P1027-60)

COATING SUPERVISOR

Recent promotions within our organization have created an opening for a man to head our research, development, and mill studies on production of machine-coated bleached board. Knowledge and experience with clay coatings are required. The person selected must have ability to lead a program in developing coating processes and new types of functional coatings. This is an outstanding opportunity for the right man. Write to:

Personnel Department
Riegel Paper Corporation
Acme, North Carolina

(P1028-60)

ACCOUNTANT

Large, new pulp and paper mill operation requires experienced accountant to head up all corporate accounting activity. Mill is now in initial design stage.

Excellent opportunity for young, (28–42) well-trained man with at least five years experience in the paper industry or comparable corporate accounting. Requires full range of accounting skills.

Forward looking, Southern-owned company offers broad opportunity, unlimited future.

All replies confidential. Address to: Industrial Relations Department

Southern Land, Timber and Pulp Corporation

795 Peachtree St., N.E., Atlanta 8, Georgia

(P1029-60)

CHEMIST—PAPER COATINGS

Eastern converter needs Chemist experienced in resinous solutions, dispersions, and emulisions. Applicant should have background in the food packaging field. Sizable plant now under construction. New and modern high-speed coating lines now on order. This 55 year old firm offers excellent opportunity and attractive compensation. Company maintains profit sharing and pension plan. Write to **P1030-60, Tappi, 360 Lexington Ave., New York 17, N. Y.**

Emulsion Chemist

Outstanding opportunity for emulsion chemist with experience and know-how in formulation and manufacturing of diffusion-transfer papers.

Kindly send resume and salary requirements to:

(P1031-60), *Tappi*, 360 Lexington Ave., New York 17, N. Y.

Continuous Calcium Hypochlorite Production at Crofton

WALTER Q. JACK and ALLAN H. DOWSLEY

This paper is presented to report on design and operation of the continuous and automatic system employed by British Columbia Forest Products, Crofton, B.C., for the production of calcium hypochlorite bleach liquor. The unique system has been in operation for more than 2 years. Advantages over the batch method are lower installation cost, reduced space requirements, reduced operating labor costs, improved stability and uniformity of hypochlorite concentration, and more complete utilization of chemicals. Bleach liquor product is semi-clarified, normally containing 0.2 to 0.4% inert solids of less than 20 μ particle size. The solids concentration is a function of reburned lime quality. Although pulp quality has not been affected, methods are being investigated to reduce the solids content to 0.2% or less. A new hypochlorite reactor of improved design was recently installed to eliminate a serious calcium carbonate scaling problem.

LATE in 1957, British Columbia Forest Products, Ltd., installed a continuous and automatic system for the production of calcium hypochlorite bleach liquor at the new kraft pulp mill at Crofton, B. C. This system is one of the first of its kind to be put into operation in North America and has been operating continuously since January, 1958. Because design and operation of this continuous system are unique, and because there are certain advantages over the batch method of preparation, considerable interest has been shown by other members of the pulp and paper industry. It is the purpose of this paper to describe details of the system design and operation, and the advantages realized over the conventional batch method.

The suitability of utilizing oxidation potential as a means of controlling the reaction of chlorine with dilute alkali to produce hypochlorite solutions was established by Pye (*1*). In 1949 the Hooker Chemical Corp. began development of a system to continuously and automatically produce calcium hypochlorite bleach liquor suitable for use in pulp bleaching operations. This system was subsequently patented in the United States and Canada (*2*, *3*). A pilot plant was constructed and demonstrated to members of the pulp and paper industry at the Hooker plant in Tacoma, Wash., in 1955. Results of this work were reported by Duncan and Rue (*4*, *5*). This was followed in October of the same year by a mill scale trial at the Camas Division of Crown Zellerbach Corp. Following these successful demonstrations, several West Coast pulp mills began planning installation of full scale mill production units based on the Hooker design. The system at the new Crofton mill was among the first of these full scale production units to begin operation. Design of this system was a joint effort by engineers of British Columbia Forest Products, H. A. Simons Ltd., and Hooker Chemical Corp.

The continuous system was chosen rather than the conventional batch method because these advantages were expected:

1. Lower installation cost. Large reaction and settling tanks would be unnecessary.
2. Reduced space requirements.
3. Reduced operating labor costs.
4. Improved stability and uniformity of bleach liquor.
5. More complete utilization of chlorine and lime.

Experience at Crofton with the continuous system has verified these expected advantages.

PROCESS DESCRIPTION

The Crofton system was designed to produce from 30 to 140 g.p.m. of bleach liquor at 35 g.p.l. available chlorine. A flow diagram of the system is shown in Fig. 1.

Lime Handling

Reburned lime, varying from 85 to 95% available lime, is slaked continuously in a DorrCo slaker. Slaked lime is diluted to approximately 10% solids and passes through the Dorr rake classifier and then to a small compartmented surge tank. The strong, hot, lime slurry is pumped from one side of the tank to a 6-in. DorrClone at 80 g.p.m. and 45 p.s.i.g. for additional classification. Rejects from the 6-in. DorrClone are removed from the system by discharging to the recausticizing slaker. Unslaked or unburned lime particles are thus recovered in the cooking liquor system. Accepts from the 6-in. DorrClone, smaller than 50μ, flow by gravity to the other side of the compartmented tank. From this side of the tank, classified slurry is pumped through a Ramen shell-and-plate heat exchanger to a 10,000 gal. storage tank. Cooling water flow to the heat exchanger is controlled to reduce the slurry temperature to about 90°F. prior to storage.

The slaker, pumps, DorrClone, and cooler are located adjacent to the recausticizing slaker at ground level, approximately 500 ft. from the strong lime slurry storage tank. The recausticizing operator starts or stops the slaker as necessary to maintain a supply of cool, classified, 10% lime slurry in storage at the bleach plant.

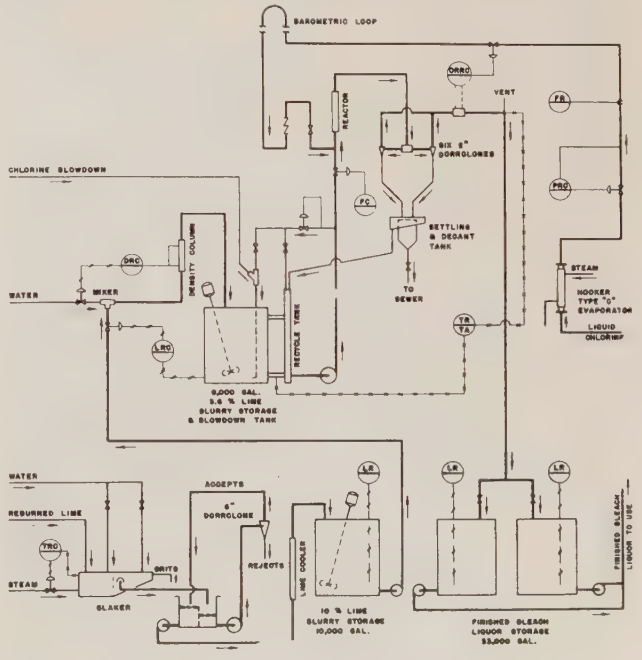

Fig. 1. Automatic calcium hypochlorite system at B. C. Forest Products, Ltd., Crofton, B. C.

WALTER Q. JACK, Technical Service Engineer, Hooker Chemical Corp., Western Chemical Div., Tacoma, Wash., and ALLAN H. DOWSLEY, Bleach Plant Chemist, British Columbia Forest Products, Ltd., Crofton, B. C.

Dilute Lime Slurry Supply

Ten per cent slurry is continuously pumped from the 10,000-gal. storage tank at ground level to an automatic dilution system on the third floor of the bleach plant directly above. A density controller regulates the flow of dilution water, and a level controller on the 8000-gal. dilute slurry storage tank regulates the flow of 10% lime slurry. The water and 10% lime slurry are mixed in a pipe coil mixer to a concentration of 3.6% $Ca(OH)_2$ at approximately 70°F.

The agitated dilute slurry storage tank provides blending, thus insuring a uniform strength slurry to supply the bleach liquor reactor. This storage also serves as a chlorine safety blowdown tank. It contains sufficient dilute alkali to absorb chlorine vented from empty tank cars or to absorb all of the chlorine contained in the chlorine handling system. Partial chlorination of the dilute slurry from venting operations does not upset bleach liquor quality, since the oxidation potential controller automatically controls chlorine flow to that which is required to obtain the desired excess alkali in the finished bleach liquor.

Dilute lime slurry is pumped to the hypochlorite reactor at a normal rate of 55 g.p.m. An additional 50 to 100 g.p.m. is recycled through a by-pass pressure control valve to the recycle tank, or through the blowdown injector on the chlorine vent system. This by-pass flow is always available to drive the blowdown injector in case emergency evacuation of the chlorine system is required. The dilute slurry storage tank and the chlorine vent system may be used to make bleach liquor batchwise; however, this has never been necessary at Crofton.

Chlorine Supply

Liquid chlorine is evaporated and the gas superheated to 150°F. One evaporator is used to supply the chlorine gas requirements to both the pulp chlorination process and the continuous bleach liquor system. High pressure gas at 100 p.s.i.g. is reduced to 75 p.s.i.g. by a pressure recorder-controller prior to flow measurement by an orifice meter and recorder. Gas flow is then controlled automatically by the oxidation potential recorder-controller. Controlled gas at about 40 p.s.i.g. flows through a barometric loop and two specially designed ball check valves to the mixing tee at the hypochlorite reactor. The barometric loop protects the chlorine system against suck-backs during shutdowns, and the check valves prevent high pressure lime from entering the chlorine piping during startup and shutdown procedures.

Reaction and Semiclarification

Dilute lime slurry and chlorine gas are mixed under pressure in a mixing tee. The unreacted mixture enters a forced circulation reactor where turbulance and a retention period of about 15 sec. are provided to complete the reaction. This reaction equipment will be described in more detail in a later section. The flow of dilute slurry to the reactor is regulated by a remote controlled flow control valve. Hypochlorite solution at 35 g.p.l. available Cl_2 and 0.8 g.p.l. excess $Ca(OH)_2$ normally flows at 55 g.p.m. and 30 p.s.i.g. to a bank of six 2-in. porcelain DorrClones for classification. Overflow product from the DorrClones at about 90°F. flows through an electrode flow cell and by gravity to one of two 17,000 gal. storage tanks on the ground level. A platinum-silver electrode pair and a Foxboro dynalog oxidation potential recorder-controller continuously measure and record oxidation potential of the product. The oxidation potential controller regulates chlorine gas flow to the reactor to maintain a constant ratio of excess lime concentration to available chlorine concentration. The reject underflow slurry from the 2-in. DorrClones, containing 1 to 3% solids, flows by gravity at a 5 g.p.m. rate into a 200-gal. settling and decanting chamber. Large unreacted lime particles and inert solids settle in this chamber and are purged to the sewer two and three times daily. Usable bleach liquor, containing smaller lime particles, is continuously decanted and recycled to the recycle tank and to the reactor. Small usable lime particles are eventually consumed during the recycle.

A temperature differential recorder with high-low alarms records the temperature rise between dilute lime slurry and finished bleach liquor. This temperature increase is due to the heat of reaction and is approximately 0.6°F. for each gram per liter of available chlorine formed. This provides a check on the accuracy of lime dilution and gives a warning signal in case a malfunction should occur in either the lime dilution system or the oxidation potential control circuit.

A low pressure alarm warns of interruption in the chlorine gas supply, and a safety shut-off, actuated by a pressure switch on the lime supply line, closes the chlorine flow control valve in case of lime flow interruption.

The reaction and semiclarifying equipment are located on the washer or operating floor directly above the lime dilution system and the dilute lime slurry storage tank. All the hypochlorite equipment is closely coupled, making it simple for the operator to check on the operation.

OPERATION AND QUALITY CONTROL

The automatic bleach liquor system at Crofton is located adjacent to the chlorine dioxide generating plant and the associated chemical handling facilities. One operator is responsible for preparation of all bleaching chemicals. All automatic controls are located in a central control room and testing laboratory. Operation of the hypochlorite system requires only a few minutes time during startup, and once in operation, little or no attention is required. Chemical tests for dilute lime slurry strength, available chlorine, and excess alkali in the finished liquor are made periodically as a check on accuracy of automatic control.

A set of Beckman platinum-silver electrodes are used to measure the oxidation potential. Calcium carbonate scale forms on the electrodes, which reduces sensitivity and slightly changes the required set point on the oxidation potential recorder-controller. The electrodes are cleaned once a day by wiping with a cloth wetted with dilute hydrochloric acid. It is then necessary to soak the electrodes in sodium hypochlorite solution for about 30 min. to restabilize them.

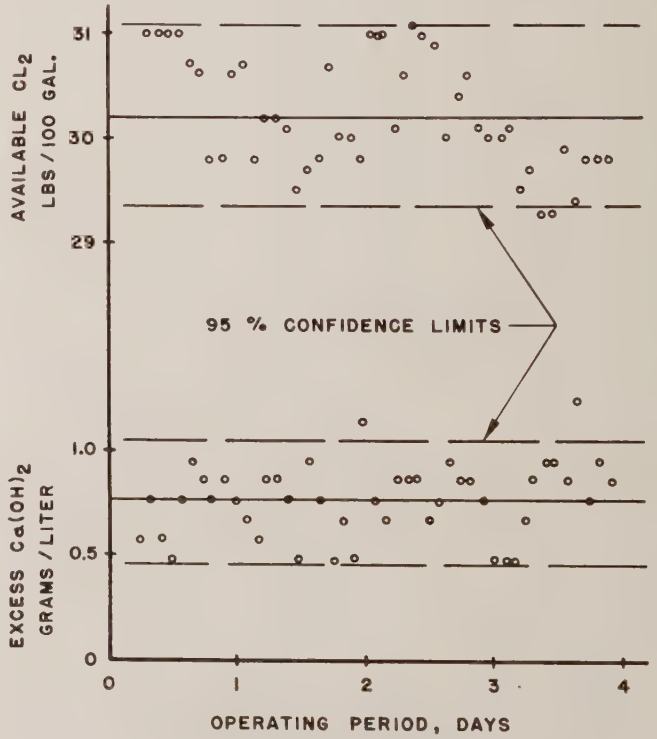

Fig. 2. Typical quality control data

A spare set of clean electrodes is always kept soaking in sodium hypochlorite solution to facilitate rapid daily changing and cleaning without upsetting subsequent control. During a 24-hr. period, it is necessary to adjust the set point on the oxidation potential controller once or twice by increasing it 5 or 10 millivolts. A control point of 620 to 650 millivolts is required to produce finished hypochlorite with less than 1 g.p.l. excess $Ca(OH)_2$.

The semiclarified finished hypochlorite solution contains about 0.3% inert solids by weight. These solids are less than 20μ particle size and give the solution a milky appearance. Solids in this low concentration and small size have had no noticeable effect on pulp quality, however, they do tend to coagulate into a light floc which partially settles in the unagitated storage tanks. This necessitates occasional cleanout of the tanks. The inert solids are composed largely of calcium and magnesium carbonates. The solids content normally varies between 0.2 and 0.4% as these impurities vary in the reburned lime supply. It has been demonstrated in pilot plant operation and in full scale operations in other mills, that solids content can be maintained at 0.2% or less if high quality lime, low in carbonate and magnesium, is employed. With high quality lime, cleanout of the hypochlorite storage tanks is seldom necessary.

At present, it does not appear that lime quality can be improved or that further preclassification of lime slurry will be effective. Therefore, other methods are being investigated to reduce the solids in the hypochlorite product.

Bleach liquor stability has been good. There has been no noticeable breakdown or loss of available chlorine during the normal 8 to 10-hr. storage period.

Uniform control of bleach liquor strength depends largely upon a uniform strength of dilute lime supply. Figure 2 shows control data charted for bleach liquor strength and excess alkali concentration. These are plots of individual tests made every 2 hr. on samples taken at the DorrClone overflow, prior to entry to the hypochlorite storage tanks. These data are typical and indicate a deviation in bleach liquor strength of $\pm 3\%$ of average or less, 95% of the time. After blending in storage, deviation in bleach liquor strength is $\pm 2\%$ or less. Excess lime tests average 0.8 g.p.l. $Ca(OH)_2$ and show a deviation of < 0.3 g.p.l. 95% of the time. Again, this deviation is reduced after blending in storage. A typical chart from the oxidation potential controller is shown in Fig. 3.

One serious maintenance problem developed shortly after the new hypochlorite system was put into operation. This was calcium carbonate scale formation in the original 2-in. PVC reaction coils and in the bleach liquor piping between the reactor and hypochlorite storage tanks. Restrictions in the reaction coils and piping eventually reduced capacity to the point that acid cleaning of the coils and pipe became necessary every few weeks. In Fig. 4 the scale formation is clearly shown in a cross-section cut of one of the reaction coil elbows. The scale formation was determined to be a function of the carbonate content of the reburned lime, being more severe at times when the carbonate content was high. Carbonate content of the lime supply normally varies between 3 and 8% as calcium carbonate. At times, cleaning of the reaction coils became necessary every few days.

This scaling problem was common in continuous calcium hypochlorite systems in other mills using small diameter pipe coil mixer-reactors. Original work had not indicated a possible scaling problem in reaction coils because operation was not prolonged and because high quality lime was used during the development work. Early attempts to solve the scaling problem were concentrated on improving lime quality and handling, which would provide a higher quality hypochlorite product. Dependable lime quality control could not be assured, and it therefore was necessary to redesign the reaction equipment to eliminate the scaling problem. The PVC pipe-coil reactor has been replaced by a new Hooker forced circulation reactor. This new reactor has been in operation for 4 months, without any indication of scaling. During this period the bleach liquor piping beyond the reactor has required cleaning but once.

REACTOR DESIGN

Prior to design and installation of the forced-circulation reactor, Hooker engineers in cooperation with the St. Helens Div. of Crown Zellerbach Corp., had field tested two prototype reactors which had proved to be scale-free during a 12-month period of operation. These two proto-types were a PVC draft tube reactor and a 3-in. saran-lined steel coil reactor. There were some disadvantages to each of these reactors and the new forced-circulation reactor was designed to incorporate only the good features of each.

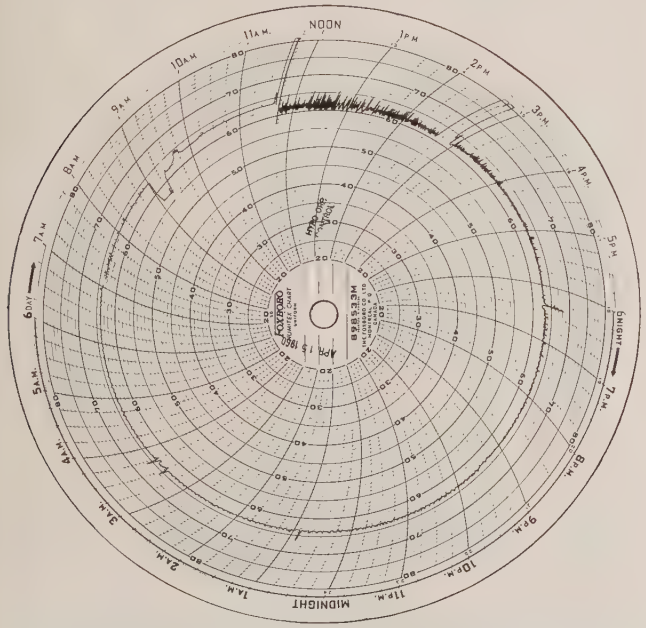

Fig. 3. Typical oxidation-potential controller chart

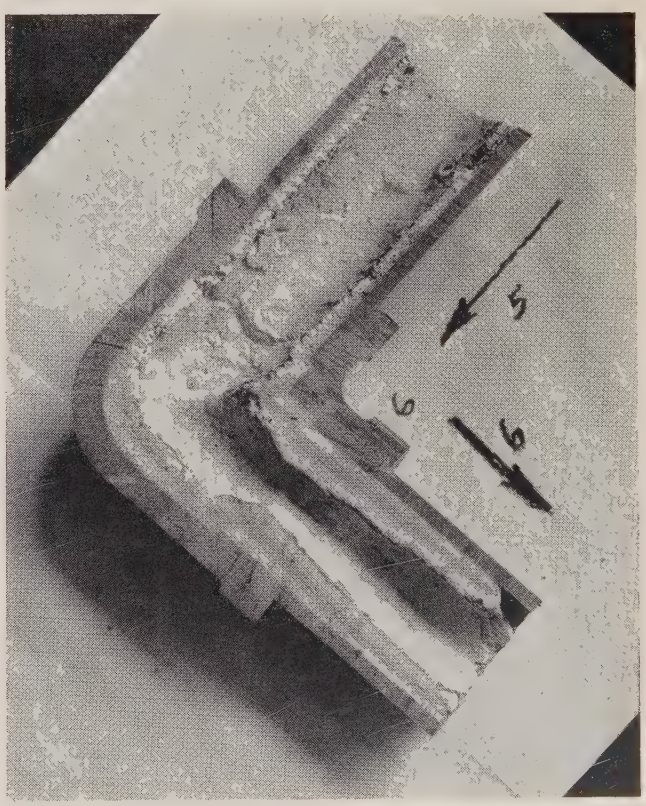

Fig. 4. Calcium carbonate scale in pipe-coil reactor elbow

Fig. 5. Forced-circulation hypochlorite reactor

The forced-circulation reactor is illustrated in Fig. 5. Chlorine gas is injected through a perforated PVC sparge nozzle and is intimately mixed with the lime slurry in the mixing tee. The mixture of lime slurry and small chlorine bubbles enters the bottom of the reactor and flows upward through the first compartment where most of the chlorine is reacted. The reaction is completed during the downward flow through the second compartment. Upward flow through the third compartment to the outlet at the top provides a safety factor to absorb excess gas in case of momentary overchlorination, or in case of a slow reacting lime slurry, and also provides additional mixing of product. Due to the volume change caused by the disappearance of gas bubbles in the first compartment, fully reacted solution recycles from the second compartment back to the first to provide a liquid flow about four times the inlet rate through the first compartment. This recirculation provides additional turbulence and mixing. A small hole through the baffle plate at the top of the first two compartments prevents gas or air binding. Should chlorine gas reach the top of the reactor due to momentary overchlorination, the gas will accumulate without leaving through the outlet and will slowly react with the liquor present. The baffled arrangement provides turbulence similar to that obtained in a pipe coil, however, the cross sectional area is sufficiently large to prevent the precipitation of scale on the walls. Retention time is only 15 sec., providing fast instrument response and excellent control.

The reactor is 5 ft. high and 14 in. diam. and operates as well at 30 g.p.m. as at 140 g.p.m. Recycle of finished bleach liquor to the reactor is not necessary for good reactor operation. Construction is of PVC reinforced with a fire-retardant polyester-fiber glass covering. The polyester reinforcing is an added safety measure for pressure operation. Under certain conditions, bleach liquor temperatures can go as high as 160°F, which is above the softening point of most PVC. This can occur if the lime cooler is by-passed or does not operate adequately, or if lime is incompletely slaked. The mixing tee at the reactor inlet is saran-lined steel. The base plate is rubber-lined steel and the top plate and outlet nozzle are PVC-lined steel.

The design features give the advantages of light weight, minimum space requirements, pressure reaction, short retention time, easy installation and maintenance, turbulent mixing, excellent resistance to corrosion, wide capacity range, nominal cost, and freedom from scaling.

Figure 6 is a photograph of the reaction equipment at Crofton. This photograph shows the forced-circulation reactor installed adjacent to the original PVC coil reactors, and shows the 2-in. DorrClones and 200-gal. settling and decant tank.

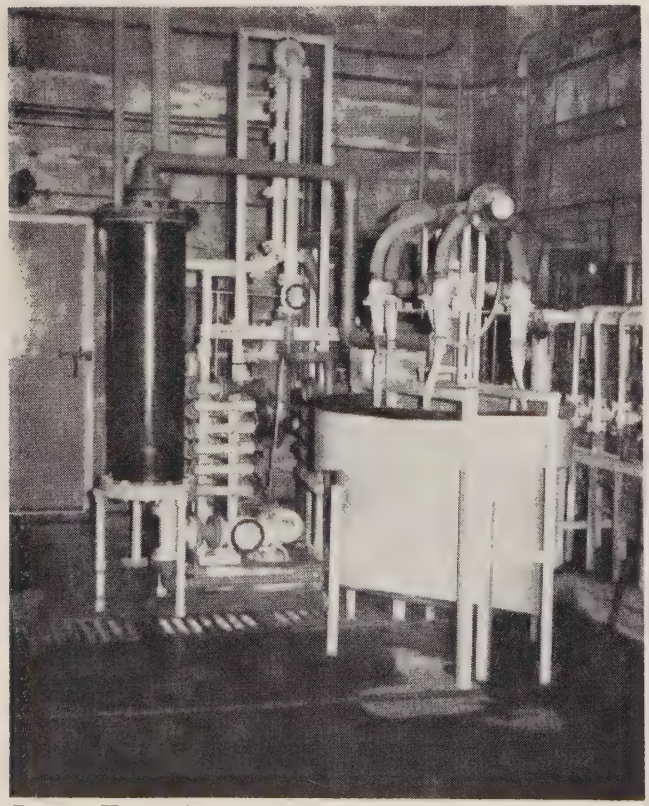

Fig. 6. Hypochlorite reaction and semiclarifying equipment

SUMMARY

The system at Crofton has been producing calcium hypochlorite bleach liquor continuously and automatically since the mill startup in January, 1958. Except for a lime scaling problem in the reactor, which has now been corrected, operation has been satisfactory and reliable. Finished bleach liquor is semiclarified and contains from 0.2 to 0.4% by weight of inert solids which are less than 20 μ particle size. Solids in this low concentration and small size have had no noticeable effect on pulp quality. Automatic control of bleach liquor strength and excess alkali have been good. A newly designed Hooker forced-circulation reactor was installed to eliminate excessive maintenance due to scale formation in the original PVC pipe-coil reactors. Advantages of the continuous system as compared with the batch method have proved to be lower installation cost, reduced space requirements, reduced operating labor costs, improved uniformity of bleach liquor, and more complete utilization of chemicals.

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The Warburg Respirometer Adapted for Use in the Industrial Laboratory

L. L. WOLFSON, P. SONG, and J. R. NELSON

One of the most important instruments in the basic research biology laboratory is the Warburg respirometer. This instrument, which measures the respiration of living cells, is used to determine the metabolic activities of bacteria. The Warburg apparatus has also proved to be a very useful tool in the industrial research laboratory. A test method has been devised for determining the bacteriostatic activity of various chemicals against certain bacteria. Test results are obtainable in one days' time against a specific organism or a group of organisms found to be associated in slime formation. The results obtained with the Warburg respirometer in the laboratory are illustrated with charts and graphs.

THE WARBURG respirometer is an instrument that has been used for many years in basic research in biological laboratories. It has been used to investigate the metabolic functions of cells, tissues, organs, enzymes and microscopic organisms, such as viruses, fungi, and bacteria. In considering various evaluation tools for the screening of chemical activity against bacteria, we felt that this instrument had many useful features and decided to adapt the method to our applied research. The data from a few of our laboratory studies are presented in this discussion.

The Warburg flasks consist of three separate sections, a sidearm, a center well, and the main body of the vessel. For these experiments, the center well contained concentrated sodium hydroxide to absorb the carbon dioxide given off by the bacteria. The change in the manometers attached to the flasks is, therefore, a measure of the oxygen taken up from the atmosphere, since the carbon dioxide is kept at the zero level. In order to compensate for atmospheric changes in the manometers, a barometer is incorporated into the Warburg.

One of our purposes was to make use of the Warburg respirometer as an instrument for testing the bacteriostatic activity of chemicals. This was accomplished by testing a standard culture of organisms against several concentrations of chemicals. The organisms chosen for the tests were *Aerobacter aerogenes*, Gram negative, aerobic, nonsporeforming rods which are often associated with slime formation. A 24-hr. nutrient broth culture of the organisms was used to prepare the cells required for one week's tests. Following incubation, the culture was centrifuged, and the supernatant liquid decanted. The cells were then resuspended in $^1/_{15}$ M phosphate buffer at a pH of 7.0, centrifuged once more, and then kept in the refrigerator in phosphate buffer for daily use.

For each test, a 5.0-ml. aliquot of the stock culture was placed in 40.0 ml. of nutrient broth, and incubated at 37°C. for 8 hr. This puts the cells into the logarithmic growth phase during which the rate of increase of cells remains constant (*1*). The cells were then washed twice in phosphate buffer and, with the use of the Klett-Summersen photometer, were adjusted to a reading of 54, which corresponds to 3×10^8 cells per milliliter. The suspension was then diluted 1:50 with phosphate buffer to give a concentration of 6×10^6 organisms per ml. One ml. of the final dilution was used as inoculum in each of the Warburg flasks.

To the sidearm of each vessel was added 0.5 ml. of the chemical to be tested, diluted to yield the desired concentration. The center well of each flask contained 0.2 ml. of 20% sodium hydroxide. At the required time, which was generally at zero hour, the chemical, kept in the sidearm, was introduced into the culture in the bottom of the flask, which was at 37°C. The manometers were then read at zero hour, at

L. L. WOLFSON, P. SONG, and J. R. NELSON, Nalco Chemical Co., Chicago, Ill.

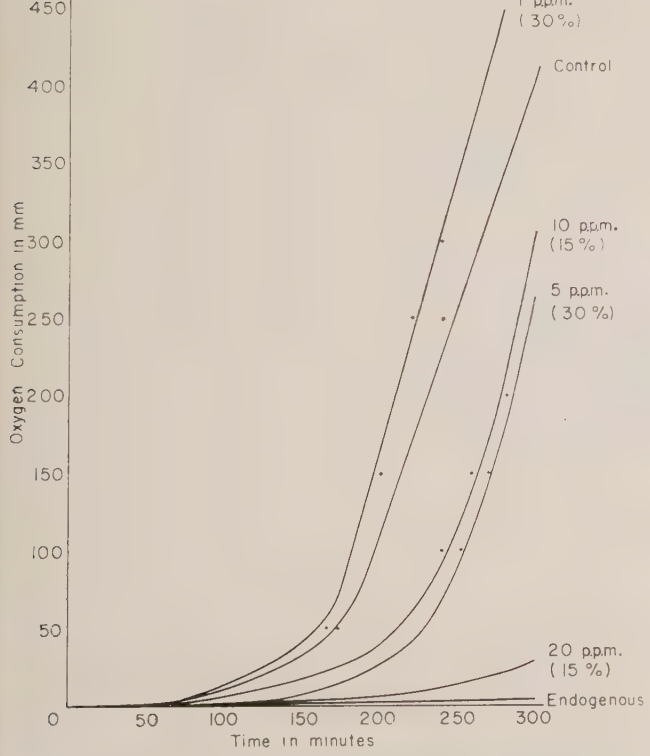

Fig. 1. Effect of 15% and 30% solutions of an amine on bacterial oxygen consumption (zero-hour additions)

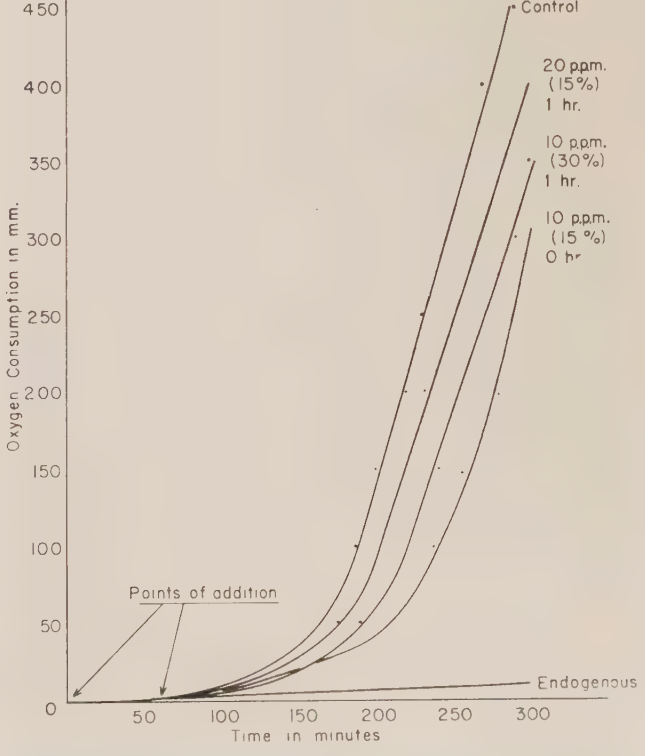

Fig. 2. Effect of 15% and 30% solutions of an amine on bacterial oxygen consumption (zero- and 1-hr. additions)

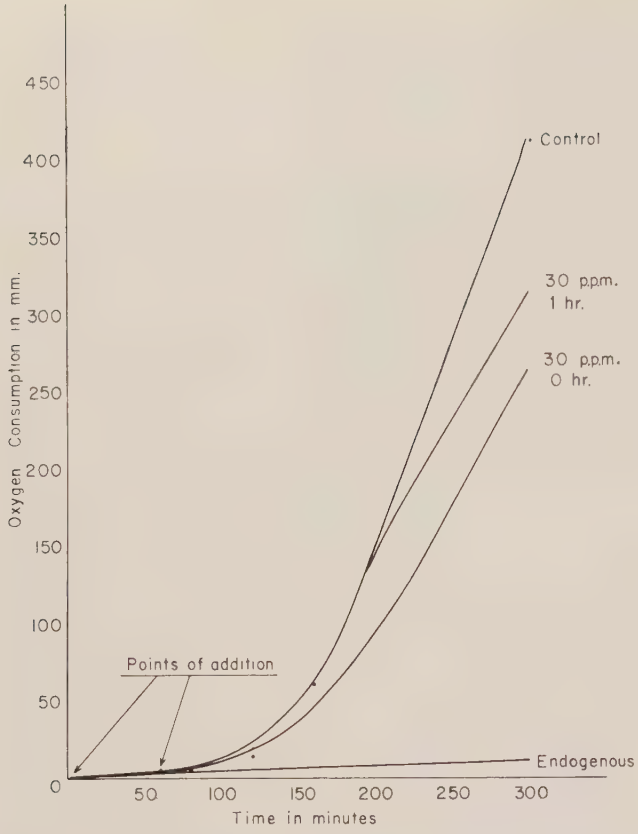

Fig. 3. Oxygen uptake of **A. aerogenes** *at zero-hr. and 1-hr. additions of a halogenated inhibitor*

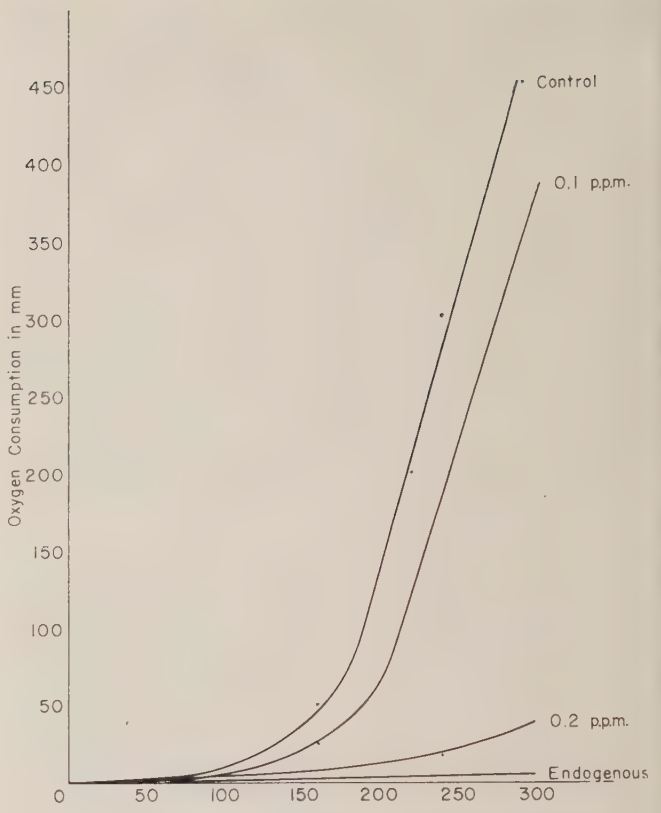

Fig. 4. Oxygen uptake of **A. aerogenes** *with addition of a bacterial inhibitor*

1 hr., and at 20-min. intervals thereafter, for 5 hr., which was the total running time of the tests.

As shown in Fig. 1, there is good reproduction in the test method. Ten p.p.m. of a 15% solution show about the same oxygen consumption as 5 p.p.m. of a 30% solution of the same chemical. The active amount of chemical is, of course, the same in both cases.

This figure also shows an apparent enhancement of bacterial activity when low dosages of an inhibitor are used. One p.p.m. of a 30% solution shows a much higher consumption of oxygen than the control which contains no inhibitor. The exact reason for this is not known, but there have been reports of this phenomenon in the literature (*2*), in which some chemicals at very low levels will actually stimulate the growth of bacteria, and yet will be inhibitory at higher levels. Perhaps this illustrates a consequence of using too low a dosage of any particular chemical.

An interesting set of results was the difference in the amount of chemical required when added after one hour instead of at zero hour. With the same chemical, as seen in Fig. 2, at zero-hour addition of 10 p.p.m. of a 15% solution, the bacteria consumed 300 mm. of oxygen, while at 10 p.p.m. of a 30% solution at 1-hr. addition, they consumed 350 mm. of oxygen. The latter addition contains twice the amount of chemical inhibitor, and yet more oxygen was consumed by the bacterial cells. The same is true of the curve showing a consumption of 400 mm. of oxygen with 20-p.p.m. of a 15% solution, which is also twice the concentration of the zero-hour addition. This may be due to the fact that there are more organisms to be inhibited after the one hour of incubation, or that once bacteria have started in the logarithmic phase of growth in the medium, they are more resistant to chemical action.

Figure 3 shows another type of chemical, a halogenated compound, where again the difference in zero hour addition versus one hour addition is pronounced.

Figure 4 shows results obtained with a new type of inhibitor. As seen here, there is good inhibition of *Aerobacter aerogenes* at 0.2 p.p.m. More information will be given on this compound as it is developed.

In summary, this set of experiments in the Warburg respirometer were inhibition tests. To date, we have not found this instrument to be satisfactory for the assay of bactericidal chemicals. It has disadvantages in that the time requirements, both in setting up the equipment and in the operation, as well as in analysis of the results, are lengthy. The cleaning of the specialized glassware requires exacting procedures much more stringent than are required with usual lab glass equipment. The tests in the Warburg do not lend themselves readily to more that a duration of 6 hr. since it requires 2 hr. per day for setting up and cleaning the equipment.

It has decided advantages over the present methods in that inhibition test results can be obtained in one day, rather than 2 to 4 days that are required in conventional tests. The results obtained with the Warburg are much more precise than present test methods. Preliminary experiments show that this instrument will enable us to determine accurately the effect of bacterial inhibitors on other organisms or possibly on mixed cultures.

We believe the respirometer has proved to be a very useful tool in the industrial laboratory particularly for the screening of bacteriostatic chemicals.

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Commercial Pulping by Total Chemical Using Elemental Sulfur in The Digester

B. D. COX, H. M. OWEN, and R. R. FULLER

An investigation was made on a commercial basis to determine the effects of sulfur addition at the digester, cooking time, total chemical, and packing of chips on pulp properties. Pulping equipment and operating procedures are described for a series of 77 cooks made under controlled conditions. Results show that relatively "soft" cooks at 27–30% sulfidity produce optimum strength properties. Both pulp permanganate number and per cent rejects decreased with increase in total chemical, cooking time, and sulfidity. A further investigation on a larger scale, showed that for a nominal chip size of $^1/_2$–$^3/_4$ in. with southern pine chips, 20–22.5% total chemical together with 27–30% sulfidity produced optimum strength. Cooking time at full pressure (100 p.s.i.) was reduced from 75 to 65 min. and chips were packed in the digesters.

SINCE the introduction of the kraft cooking process, the search for optimum pulp yield and strength properties has generated a wealth of data concerning reaction mechanisms, chip size, sulfidity, and chemical-to-wood ratio. Variations in raw material, pulping equipment, and type of pulp desired have produced varying conclusions. Regarding the interaction of sodium hydroxide and elemental sulfur in white liquior, Kress (*4*) has presented the following reactions:

Primary reaction: $6NaOH + 4S \rightarrow 2Na_2S + Na_2S_2O_3 + 3H_2O$

Secondary reaction: $6NaOH + nS \rightarrow 2Na_2S\left(\frac{n-2}{2}\right) + Na_2S_2O_3 + 3H_2O$

His experiments showed that at pulping temperature and pressure, sodium sulfide and thiosulfate concentrations increased with addition of sulfur. The ratio of sulfide to thiosulfate did not change. Kress also suggested that sodium thiosulfate played a negligible part in the pulping action. In an investigation of polysulfide pulping, Peckham and May (*5*) determined that the cooks rich in "active" sulfur produced a 2% increase in yield with pulp strength properties slightly inferior to those of regular kraft pulps.

Thus, it is evident that addition of sulfur to white liquor plays an effective part in the kraft pulping process.

With regard to the optimum sulfur content or sulfidity of white liquor, Hanson (*3*) has stated that optimum sulfidity depends primarily upon per cent total chemical and is independent of liquor concentration. For a cook having 20% total chemical (based on dry wood), optimum sulfidity is 30%. Previous work by Bray, Martin, and Schwartz (*1*) on longleaf pine shows that at a per cent total chemical to wood of 18.75 to 20%, the yield of screened pulp passes through a maximum. Less than 18.75% results in increased screenings; more than 20% causes a greater destruction of cellulose than lignin removal. The commercial cooking trials of Chesley and Gilmont (*2*) indicate no detectable difference in pulp quality when sulfidity is reduced from 30 to 20%.

Thus it would appear from previous work that optimum, sulfidity in the kraft process, as well as per cent total chemical, would depend upon particular factors encountered at the mill. From the work of Robertson (*6*) and Suttle (*7*), chip size is one of these factors.

With this literature as a background, it was decided to investigate on a commercial basis the effects of total chemical, sulfidity, and cooking time upon the kraft process at Gulf States Paper Corp.'s Tuscaloosa plant. The specific goals of the investigation were:

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1. To evaluate the effects of cooking time, total chemical, and packing of chips upon pulp properties.
2. To determine the effects upon pulp quality when elemental sulfur is added at the digesters.
3. To develop improved pulping procedures and judge their effectiveness by comparison with normal pulp output and quality.

EQUIPMENT AND PROCEDURES

Pulping equipment consisted of eight batch-type digesters utilizing injected steam to attain a cooking pressure of 100 p.s.i. The digester charge is circulated by action of the steam only and gas-off operation is manual—approximately constant throughout the pressure buildup and cooking phases. Four of the digesters, designated "A" side, have a volume of 2700 cu. ft. each and discharge through a cyclone to a circle of 12 diffusers. The remaining four digesters, designated "B" side, have a volume of 2800 cu. ft. each and discharge to a blow tank—thence to continuous countercurrent pulp washers.

In order to evaluate the effects of process variables upon pulp properties and also use a minimum of production equipment, the first phases of the investigation were made using only one of the "A" side digesters. Thus we were able to vary operating conditions over a considerable range without disrupting production schedules. Pulp samples were taken directly from the digester or from the digester blow line. During July, 1958, 77 cooks were made at this digester under the following controlled conditions:

Variable	*Levels at which controlled*	*Means of control*
Liquor sulfidity, %	18–20, 23–25, 27–30	Adding sulfur[a] at the digester prior to liquor addition.
Total chemical per cook, lb.	6500, 6800, 7200, 7600, 8000	Metering white liquor after determining lb. of total chemical/gal. (as NaOH, etc.) from ABC tests.
Total liquor vol., gal.	7500	Metering black liquor.
Quantity of chips per cook, lb.	Unpacked = 62,000 Packed = 68,000 Av. moisture = 50%	Unpacked chip charge consisted of filling digester to top prior to liquor addition. After liquor addition had lowered chip level, chips were packed simply by adding extra chips to fill the space created.
Cooking time at full pressure, min.	60, 80, 100	Sampling and blowing cooks at the prescribed time periods.

[a] NOTE: The quantity of sulfur to be added was calculated from white liquor ABC tests as the theoretical amount necessary to increase sulfur concentration to the desired level. Although this concentration is expressed as Na_2S or Na_2O, it is highly improbable that all the added sulfur became bound as Na_2S. No means were available to determine the extent or nature of this combination. However, for purposes of discussion, the term "sulfidity" will be used. Caustic and sodium carbonate concentration as Na_2O in the white liquor were 0.95–1.00 lb./gal. and 0.12–0.16 lb./gal., respectively.

Throughout the course of these cooks, southern pine chips less than 30 days old and having $^1/_2$–$^3/_4$ in. nominal chip size were used. In order to obtain data representative of specific operating conditions and also to avoid an excessive backlog of pulp samples, two to four cooks were made at each level of control and their pulp samples were composited before tests were made. Laboratory tests included:

Chip size and moisture content
White liquor ABC tests and $AgNO_3$ sulfidity
Pulp permanganate number and per cent rejects
Pulp handsheet strength tests after refining in a laboratory Valley beater to 300 ml. C.S. freeness.

From these results several plots were made to determine the effects of process variables upon the resulting pulp.

Table I Data for Experimental Cooks at No. 3 Digester

Adjusted % solidity	Total chemical, lb	Chips pressed?	Ex cook numbers	Chip size: % Chips retained on screen size: 2"	1"	3/4"	1/2"	5/16"	Fines	Chip moisture, %	Weak black liquor: Sp gr 68°F	Total solids, g/l	Alkali as Na2O, g/l	Temp, °C	White liquor tests: Before sulfur added at digesters: A	B	C	% ABC sulfur	% AgNO3 sulfur	Weak black liquor, g/l	wh. liquor, g/l	Pulp KMnO4 no. av.: 60 min cook dig bline	80 min cook dig bline	100 min cook dig bline	% Rejects av.: 60 min cook dig bline	80 min cook dig bline	100 min cook dig bline	Av TAPPI burst factor at 300 ml free: 60 min cook dig	80 min cook dig.	100 min cook dig.	Av TAPPI tear factor at 300 ml free: 60 min cook dig.	80 min cook dig.	100 min cook dig.	Av TAPPI breaking length (km), 300 ml free: 60 min cook dig	80 min cook dig	100 min cook dig	Av TAPPI fold % at 300 ml free: 60 min cook dig.	80 min cook dig.	100 min cook dig.
18-20	6500	NO	A	0	28.0	22.9	25.2	15.7	7.3	59.5	.007	225	.58	95	T2	B1	18.9	16.0	2360	5.40	28.3	24.7	25.4	21.5	18.8	11.5	65	65	69	125	130	130	132	13.5	13.7	4200	3500	4600
			2A	1.3	20.0	20.5	29.0	19.5	9.5	55.8	1.01	324	1.23	95	T2	B1	18.9	14.0	2360	5.40			23.8			.02												
			3	.8	2.2	22.7	26.4	9.0	9.8	52.0	1.016	432	1.09	95	T0	B0	21.0	14.8	2.95	5.56	26.0	25.4	24.8	19.3	10.2	5.4												
18-20	6800	NO	6	0	6.4	15.1	30.5	3.1	17.1	54.2	1.016	477	.94	95	T0	B0	21.0	16.2	2190	5310							68	67	70	145	140	150	14.4	13.8	15.5	3500	4300	3800
			8	.6	28.7	18.6	24.9	17.4	8.7	50.4			.85	98	T2	B2	20.4		2370	5310			21.7			4.7												
18-20	7200	NO	9	0	23.3	22.3	27.1	18.2	9	54.1	1.016	373	1.17	100	T4	B4	20.0		2190	5310	23.9	24.3	23.8	14.4	9.7	5.2	66	71	72	150	140	150	12.6	14.6	14.6	6400	4500	3500
			0	0	4.3	8.2	29.7	25.1	27	48.5	.022	513	1.75	100	T4	B4	20.0	17.8	2190	5310			19.8			4.0												
18-20	7500	NO	3		22.4	23.9	26.8	16.8	9.0		.026	434	1.28	96	T3	B2	18.8	20.2	1620	5880	24.5	22.4	19.4	15.3	6.0	5.3	68	72	73	130	145	140	14.5	14.7	15.3	4500	4000	5500
			14	0	8.2	22.2	29.6	9.6	10.4	52.6	.020	406	1.14	96	T3	B2	18.8	15.7	1620	5880			19.0			5.3												
18-20	8000	NO	7	0	5.7	18.9	3	24.5	9.9	51.0	.030	499	1.53	100	T4	B4	20.0	17.2	1600	5900	23.7	20.5	19.5	11.0	6.0	4.5	61	65	68	140	130	140	13.5	14.0	14.4	3000	3100	4500
			8	0	5	16.0	32.0	28.4	12.1	5.1	.020	495		99	T4	B4	20.2	18.6	1500	6000																	
18-20	6800	YES	21	.8	25.3	21.2	25.8	8.6	10.5	53.4	.016	367	1.04	97	T2	B2	20.6	18.2	2330	5170	27.2	26.0	24.2	25.6	18.0	13.2	63	64	63	140	155	155	13.0	14.0	14.2	3000	4000	4700
			22	0	4.4	17.8	30.0	26.2	11.6	53.7	1.046	618	1.38	0.	T4	B3	17.9	15.0	2650	4950			23.8			12.3												
18-20	7500	YES	25	0	16.0	23.4	29.3	21.3	9.9		1.029	540	1.47	96	T3	B2	18.7	15.5	1620	5880	25.4	22.8	21.2	14.0	9.2	10.4	65	70	73	150	150	170	4.1	14.2	15.0	5200	4500	4000
			29	0	5.5	19.3	32.4	22.1	10.8	52.4	.025	610	.84	101	T4	B3	17.9	18.0	1680	5820	24.4	23.0	20.2	14.0	7.6	5.6	65	66	65	150	145	155	14.0	14.0	4.5	3300	3400	3000
18-20	8000	YES	30	.7	1	16.8	33.5	25.4	11.7	52.2	1.017	420	1.01	98	T1	B1	20.4	13.7	1500	6000																		
23-25	6500	NO	33	2.5	21.2	22	26.3	8.0	10.6	51.5	1.016	337		98	T1	B2	22.4	20.6	2600	4900																		
			34	.2	20.8	19.7	27.8	9.6	10.9	52.7	1.019	.296	.96	96	T1	B2	23.0	22.0	2450	5050	28.0	27.0	25.4	20.6	15.0	10.	63	66	70	130	150	140	14.0	14.0	14.4	8200	2000	7500
			35	0	2.2	4.7	24.0	26.5	22.8	50.8	1.025	432	1.09	96	T1	B2	23.0	21.6	2450	5050																		
			36	4	17.6	18.3	27.2	21.0	15.5	55.1	1.019	.341	T4	90	66	T7	24.4	22.2	2120	5380		23.0			10.0													
			37	..	1.8	6.0	33.4	28.0	10.7	51.8	1.016	536	9.1	90	67	T8	24.4	21.2	1860	5640																		
23-25	6800	NO	38	.7	20.2	21.1	28.8	20.0	9	54.9	1.019	320	.0.	96	T1	B2	23.0	21.9	2230	5270	25.9	23.8	20.8	14.8	9.1	7.5	71	72	75	130	135	130	14.5	14.8	16.2	2000	3000	2800
			39	1.2	23.8	21.7	25.5	7.8	10.0	53.2	1.018	361	1.32	97	T1	B2	22.7	21.7	2330	5170		19.5			9.2													
			40								.023	443	1.28	97	T1	B2	22.7	21.9	2330	5170																		
			41								1.013	299	.88	99	T1	B3	22.8	21.6	2150	5350																		
23-25	7200	NO	42	0	0.3	17.8	35.4	25.2	1.2	53.	1.017		.86	100	T2	B4	24.0	22.0	2190	5310	24.0	22.8	20.5	14.3	9.0	6.6	70	71	74	155	160	165	14.3	14.8	15.1	4800	5000	6700
			43	0	9.6	19.7	34.0	25.4	1.4	53.0	1.002	.394	1.12	100	T2	B5	26.0	22.4	2150	5350																		
			44	0	12.7	17.4	33.0	26.2	10.7	55.2	1.019	.362	1.05	100	T2	B4	24.0	22.2	2190	5310		22.3			7.5													
23-25	7500	NO	45	0	9.3	20.1	28.8	21.8	9.9	51.6	1.016	332	8.5	99	T3	B4	22.2	21.4	1880	5620																		
			46	0	8.3	19.7	28.4	22.1	11.5	5.2	1.012	266	8.5	99	T2	B4	24.2	22.6	1880	5620	24.0	22.5	19.4	15.0	6.9	5.8	66	71	69	150	145	175	14.5	16.2	15.5	4500	3000	6500
			47	.8	8.0	20.7	27.5	20.2	2.6	51.5	1.010	265	1.17	99	T2	B4	24.2	22.0	1880	5620																		
			48	.8	23.1	20.4	25.8	18.8	11.5	47.3	1.027	400	1.18	98	T2	B3	22.5	21.5	1840	5660			18.8			5.3												
23-25	8000	NO	49	.1	20.0	20.7	27.9	19.1	12.1	48.0	1.016	405	8.9	99	T2	B3	22.2	24.2	1550	6950																		
			50	0	17.2	20.0	31.1	22.1	9.7	51.3	1.018	.375		98	T2	B3	22.5	21.2	1460	6040	23.2	20.0	17.6	9.6	4.0	3.9	65	68	69	155	140	160	14.6	15.2	14.8	9000	3200	5700
			51	.6	15.8	19.2	28.8	23.3	12.3	47.8	1.009	.357	7.0	97	T2	B3	22.7	—	1570	6130																		
			52	0	16.6	18.7	30.5	22.5	11.6	50.0	1.009	224	T4	98	T1	B1	20.3	19.6	1500	6000	19.0			7.9														
23-25	6800	YES	53	1.4	0.3	16.8	29.0	27.9	14.5	50.2	1.009	216	1.03	98	T1	B1	20.3	19.2	2410	5090																		
			54	0	22.9	19.9	27.6	20.2	9.5	51.5	1.014	302	1.20	98	T1	B1	20.3	19.4	2410	5090	27.4	25.0	24.0	22.2	9.1	13.5	67	67	68	145	155	160	14.2	14.6	14.9	4000	5700	5000
			55	2.9	20.2	20.6	29.6	26.7	—	53.4	1.015	.313	.81	98	69	B0	22.5	19.6	2390	5140																		
			56	3	22.7	19.8	26.0	17.8	14.3	55.1			.99	99	T4	B5	22.3	16.3	2410	5090			24.2			10.9												
23-25	7500	YES	57	0	13.1	16.5	27.4	25.0	17.9	52.2	1.014	.278	7.6	98	69	B0	22.5	19.2	1860	5640																		
			58	0	8.0	15.0	28.0	26.8	23.4	48.2	1.012	.280	.91	98	69	B0	22.5	18.6	1860	5640	23.8	21.1	20.3	10.0	8.4	5.2	71	70	72	165	170	170	15.0	14.7	14.1	5800	6000	6500
			59	.8	20.3	19.0	28.6	19.6	11.7	52.1	1.003	.236	9.7	102	T5	B7	23.5	18.7	2050	5450																		
			60	1.6	11.1	17.8	32.4	24.8	12.3	54.9	1.015	292	1.17	99	T2	B2	20.2	19.6	1930	5870		20.7			5.8													
23-25	8000	YES	61	1.6	18.6	21.0	25.4	9.8	12.6	51.7	1.00	235	.93	102	T5	B7	23.5	17.3	1680	5820																		
			62	1.1	13.0	20.2	31.7	23.0	.0	51.5	1.01.	250	6.8	103	T5	B7	23.3	17.0	1720	5780	23.4	20.5	20.0	9.5	6.9	4.1	63	64	64	160	160	170	13.3	14.0	14.2	3000	5300	7300
			63	0	14.3	17.1	30.7	25.4	12.6	55.0	1.015	.265	8.5	100	T4	B5	22.0	19.0	1550	5950																		
			64	0	17.8	16.0	28.5	23.8	13.8	50.6	1.009	211	6.8	100	T4	B5	22.0	15.2	1660	5950			20.2			4.7												
28-30	6500	NO	65	.8	23.8	21.4	28.2	17.2	10.6	54.	0.9	345	.64	93	68	B0	25.8	20.7	2280	5220	25.7	24.6	24.1	19.1	14.1	9.0	68	69	76	55	150	170	14.7	15.8	15.0	5000	5500	4500
			66	6	14.2	19.7	32.0	23.2	0.3	54.8	1.022	.427	.87	97	T1	B2	22.7	21.2	2560	4940																		
			67								1.006	207	T4	97	T2	B4	24.8	22.7	2520	5000																		
28-30	6800	NO	69	1.4	4.1	18.9	30.2	23.7	1.8	51.9	1.009	.210	.61	100	T4	B5	22.0	17.6	2440	5060																		
			70	0	9.1	16.9	25.0	23.2	15.8	54.7			.89	98	T2	B4	24.6	21.0	2330	5170	24.4	23.0	23.4	17.0	10.0	7.9	69	68	71	145	170	185	14.1	14.9	15.4	4200	8200	5200
			71	.3	34.7	22.1	22.0	13.7	7.3	51.7			.95	98	T1	B2	22.5	20.6	2310	5130																		
			72	0	23.0	19.7	27.4	19.4	10.5	55.2			.67	1.01	T2	B3	21.8	21.2	2550	4950		23.7			9.3													
28-30	7200	NO	73	0	22.1	19.6	28.4	19.2	10.6	51.0			.98	99	T2	B3	22.2	22.3	2150	5350																		
			74	0	25.3	25.8	26.0	16.4	8.4	52.0			.84	99	T2	B3	22.2	15.3	2150	5350	24.0	23.0	20.0	11.2	10.2	6.8	65	71	72	160	180	180	14.5	14.7	16.0	8200	9000	6000
			75	.2	21.2	21.8	26.7	19.4	10.5	48.1	1.012	.285	.86	99	T2	B3	22.2	17.5	2150	5350																		
			76	0	20.8	18.9	27.1	21.0	12.3	49.0			.71	99	T2	B3	22.2	22.0	2150	5350			19.0			6.4												
28-30	7500	NO	77	9	29.5	25.4	26.2	14.4	5.6	48.6			.79	101	T3	B5	23.8	18.0	2010	5490																		
			78	0	22.4	20.9	27.8	19.4	9.5	47.3			.80	99	T3	B4	22.2	21.3	1880	5620	22.3	21.0	19.8	12.4	6.1	3.1	68	72	73	70	185	90	14.2	15.6	15.8	3500	6000	6500
			79	0	13.5	19.1	33.1	24.8	9.4	49.4			.83	99	T3	B4	22.2	16.3	1880	5620																		
			80	0	23.9	22.6	27.9	16.8	8.8	52.5	1.015	.363	1.12	99	T3	B6	24.2	19.2	1880	5620			20.0			3.3												
28-30	8000	NO	8	0	20.4	23.4	28.4	18.4	9.5	50.6			1.13	99	T3	B5	24.2		1500	6040																		
			88	0	21.4	22.2	25.2	20.6	10.6	5.3	1.016	.341	1.07	100	T3	B4	25.0	9.0	1600	5900	22.6	19.4	17.6	7.5	2.9	3.0	66	68	67	165	175	170	14.5	14.2	15.2	5200	4700	4700
			84	0	16.7	21.3	31.1	20.6	10.3	51.9	1.015	.313	.96	100	T3	B4	22.0		1600	5900			7.5			3.6												
28-30	6800	YES	85	0	19.9	22.0	28.6	19.1	10.3	50.4	1.020	.824	.23	98	T2	B3	22.5	9.5	2310	5130	26.2	26.0	23.2	19.6	13.5	10.6	66	69	71	160	185	175	13.7	14.8	15.0	3200	2600	5500
			86	0	22.2	20.1	27.1	20.4	10.4	49.7	1.018	.443	1.36	98	T2	B3	22.5	9.3	2310	5130																		
			88	0	14.1	18.4	30.2	26.6	11.7	52.4	1.020	.430	1.21	99	T3	B4	22.2	16.3	2410	6090			22.4			8.1												
28-30	7500	YES	89	0	25.8	23.4	25.8	17.4	7.5	47.8	1.020	.401	1.20	99	T3	B4	22.2	14.0	1880	5620																		
			90	0	20.6	23.6	27.5	19.2	9.2	50.3	1.012	.366	1.10	101	T3	B4	21.8	15.0	2010	5490	23.6	21.6	21.	3.7	6.0	5.9	68	71	74	80	185	190	14.4	15.2	6.4	3000	4500	4100
			92	1.2	16.1	19.8	30.8	22.5	9.5	51.8	1.018	.487	1.07	101	T3	B4	21.8	14.6	2010	5490			21.0			4.8												
28-30	800	YES	93	0	13.7	16.9	32.1	26.5	10.7	52.6	1.014	322	1.02	99	T4	B5	22.2	15.8	1500	6000	22.6	21.8	19.2	9.0	5.7	3.3	67	69	70	155	170	185	14.1	15.0	15.5	4000	3000	4100
			94	0	12.5	16.1	27.2	25.7	18.5	49.3	1.029	.568	1.62	10.	T3	B3	19.8	17.3	1680	5820			19.4			3.0												

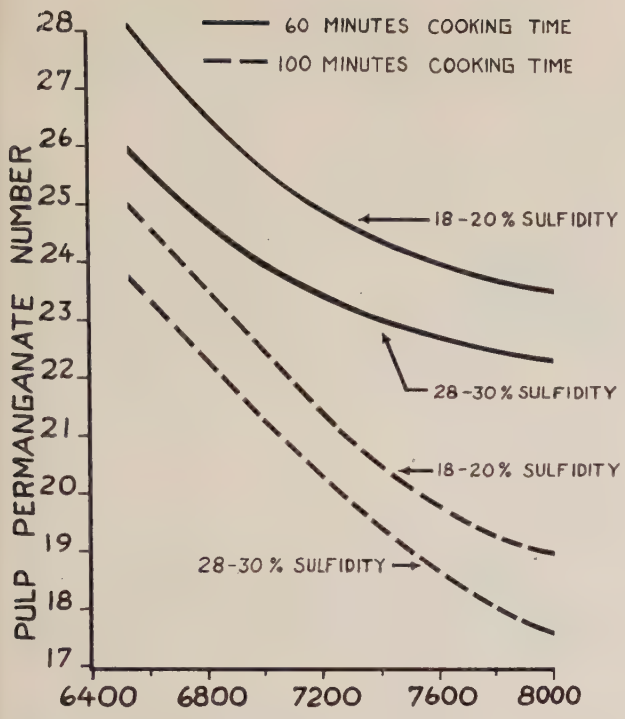


Fig. 1. Chips not packed.

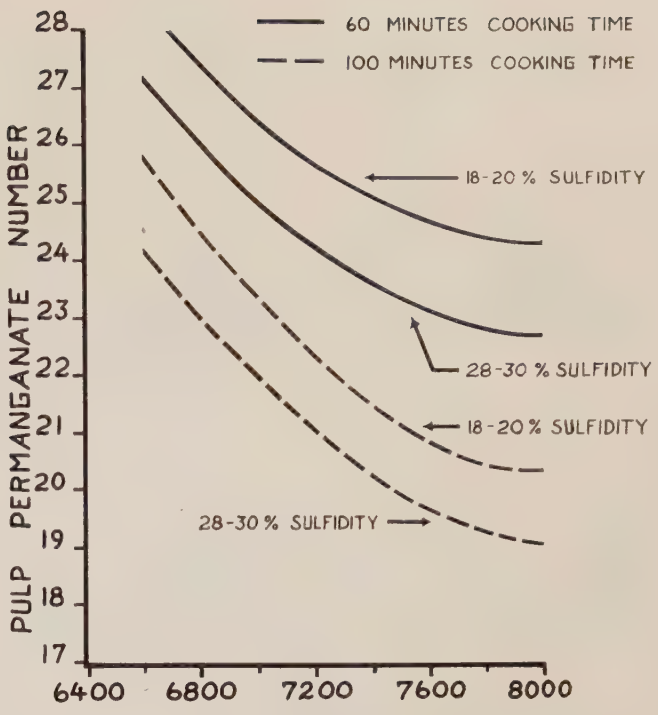


Fig. 2. Chips packed.

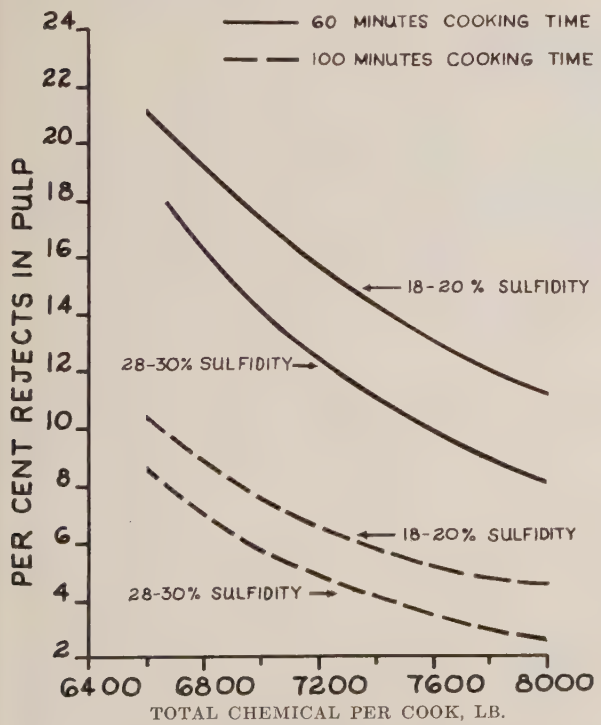
Figs. 1 and 2. Pulp permanganate number vs. pounds of total chemical per cook

The most significant of these effects is presented in Figures 1–7. Basic data for these cooks are presented in Table I.

DISCUSSION

Although the results of Figs. 1 and 2 may be anticipated possibly, the degree to which the process variables affect pulp permanganate number during a commercial cook is significant. The variables having greatest effect upon permanganate number are total chemical per cook and cooking time (at full pressure). Thus as total chemical increases from 6600 to 8000 ib. per cook or from 19 to 26%, there is a 3 to 6 point drop in permanganate number. It is noteworthy that all four variables shown are interdependent in their effect upon permanganate number.

As was previously mentioned, the complete conversion of the added sulfur to Na_2S is highly improbable. This assumption is substantiated by the work of Kress (*4*). Nevertheless, an increase in sulfur content as shown does decrease permanganate number by 1 to $1^1/_2$ points. Thus at high "sulfidity," less white liquor is needed to achieve the same permanganate number.



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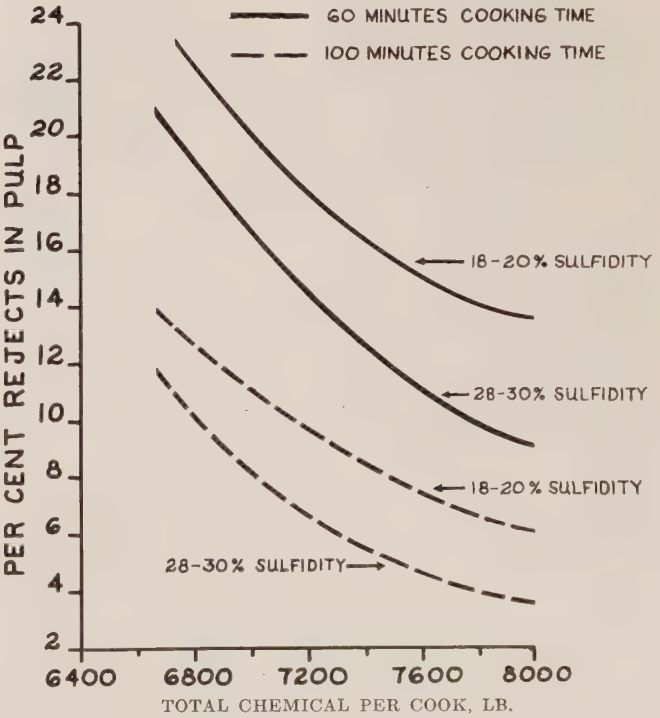

Fig. 4. Chips packed.

Figs. 3 and 4. Per cent rejects in pulp vs. pounds of total chemical per cook

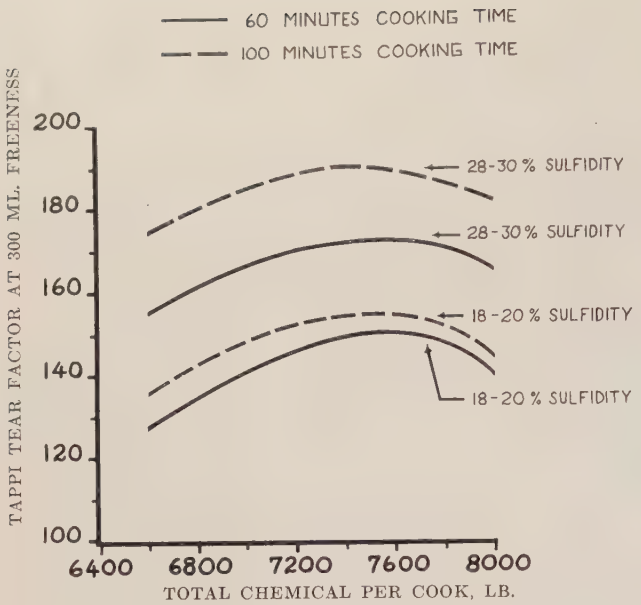

Fig. 5. TAPPI tear factor vs. pounds of total chemical per cook

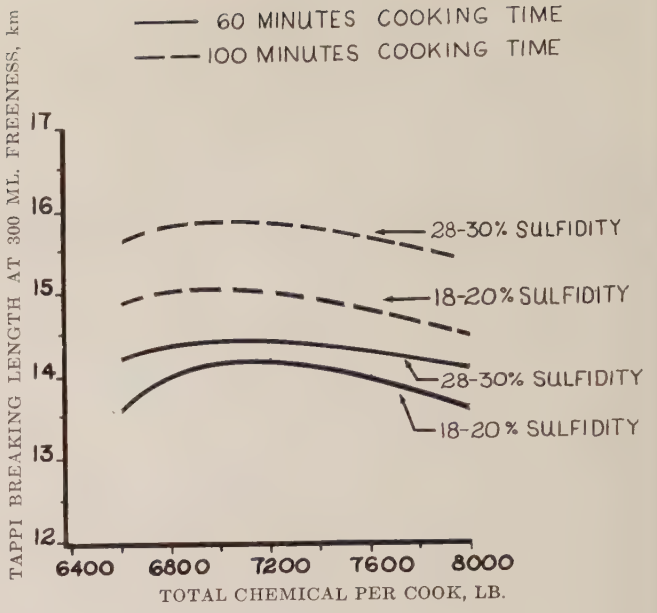

Fig. 7. TAPPI breaking length vs. pounds of total chemical per cook

When chips are packed in the digester, the resulting permanganate number is approximately one point higher--all other conditions being the same.

With reference to Figs. 3 and 4, the effects of the variables shown upon per cent rejects in pulp is very similar to their effects upon pulp permanganate number. Again, we see total chemical and cooking time producing the major changes in the pulp property and in the same direction. Sulfidity and chip packing exhibit the same trends as before. The very similarity of these effects suggests a direct correlation between the two pulp properties under given cooking conditions. Indeed under the controlled conditions of the trial, a correlation was established. In later trials it was found that this correlation depended not only on the four variables illustrated but also on chip size and age. Nevertheless it is significant that the variables illustrated affect pulp per cent rejects and permanganate number in the same manner.

In contrast to those on pulp permanganate number and per cent rejects, the effects of process variables upon strength changes with the strength property in question. In Fig. 5, showing the effects upon tear, sulfidity is the major variable. This particular phenomenon is quite noteworthy since it indicates that within the conditions tested, high sulfur content

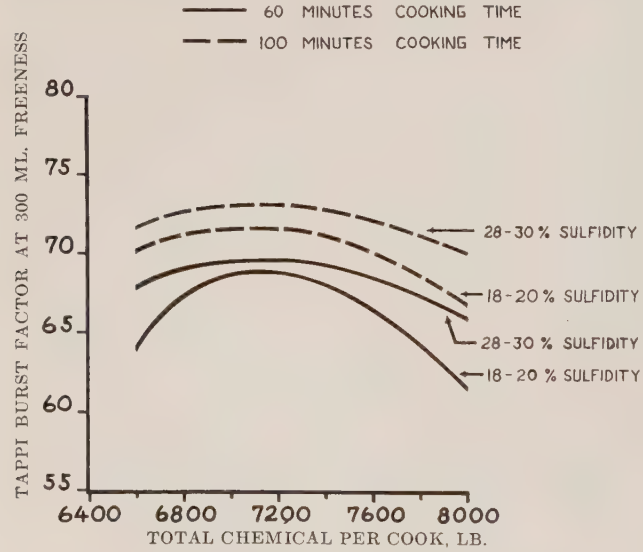

Fig. 6. TAPPI burst factor vs. pounds of total chemical per cook

engenders high tear strength whether the cook be relatively "hard" or "soft." Indeed a high-sulfur, "soft" cook offers the highest tear strength of any of the conditions encountered. Thus tear strength is also favored by the longer cooking time. Increase in total chemical increases tear until 7000 lb. (22.6 %) per cook are reached; at which point tear begins to drop off with further chemical. The curves for packed chips, similar to the ones indicated (unpacked), reached a maximum tear value at 7600 lb. (22.3%) total chemical per cook.

When the effects of these variables upon burst strength are plotted (see Fig. 6), cooking time as well as sulfidity are the major variables. Here both the 100-min. cook curves offer increased burst; the highest strength associated with high-sulfur, relatively "soft" cooks. These same trends are exhibited in Fig. 7 illustrating the effects upon breaking length. For both Figs. 6 and 7, chip packing had little effect upon strength properties such that packed and unpacked chip curves are very similar.

In review of the trends exhibited by Figs. 1–7, indications are that a relatively soft cook at high sulfur content not only produces lower permanganate number and per cent rejects, but also higher strength properties. The effects upon digester capability were not yet known. In further investigation of these trends and their applicability to plant-scale operations, two other experimental digester runs were made. The first of these runs took place in November, 1958, and consisted of 3-days' operation of all four "B" digesters. Experimental operating conditions as well as normal cooking procedures are listed as follows:

	Experimental cooks	*Normal cooks*
White liquor sulfidity, %	27–30 (by adding S at digesters)	20–24
Total chemical per cook, lb.	8000	6600–6800
Total liquor volume, gal.	7900	8000
Chips per cook, lb.	71,000—packed (50% moisture)	68,000 (50% moisture)
Total chemical, %	22.5	19–20
Cooking time at 100 p.s.i., min.	75	75
Type wood used	Southern pine; chips less than 30 days old.	Southern pine; chips less than 30 days old.

NOTE: During the experimental cooks, nominal chip size was 3/4 to 1 in. rather than 1/2 to 3/4 in., owing to modification of chipping facilities.

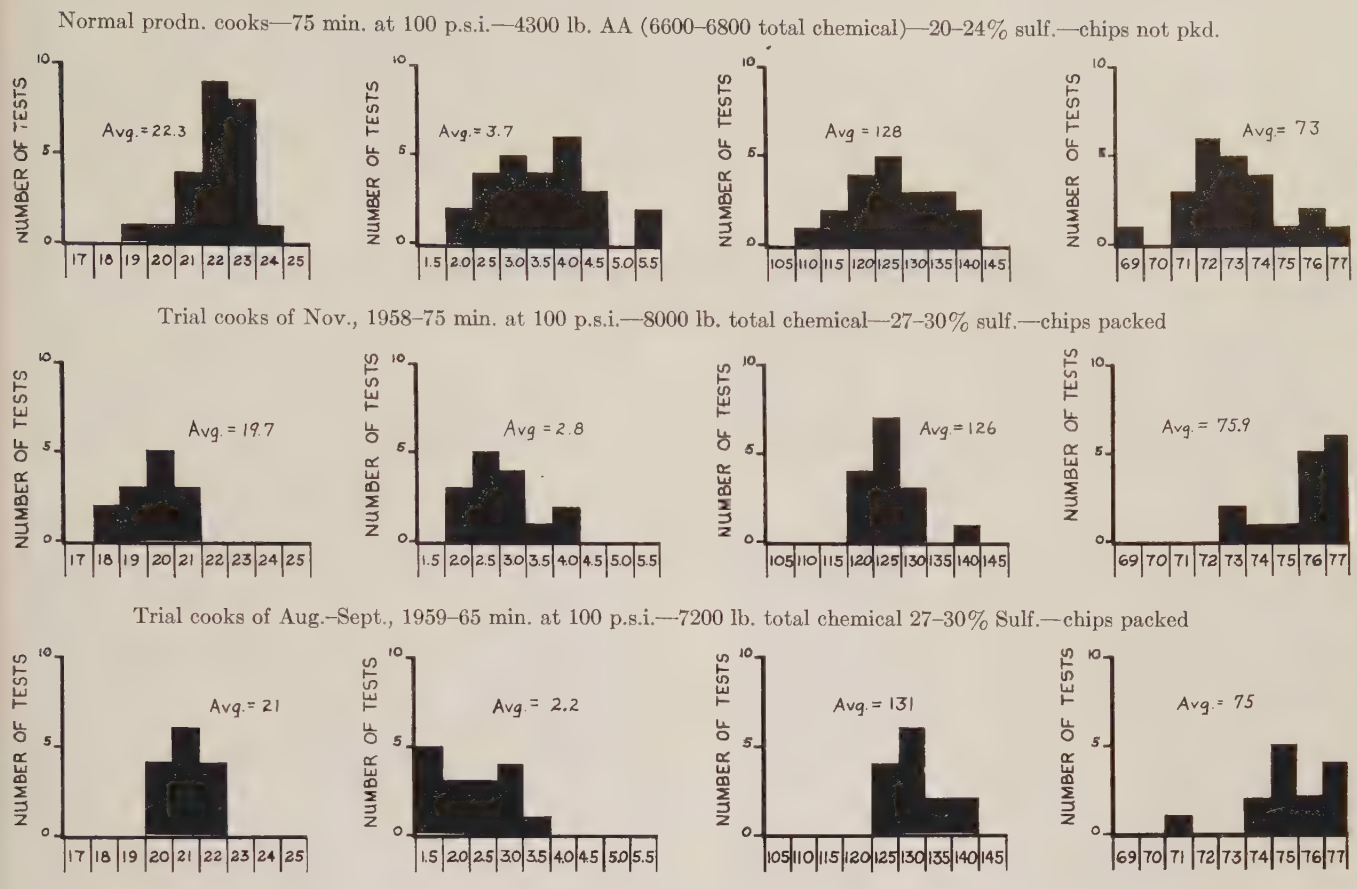
Pulp permanganate number
Per cent rejects in pulp
TAPPI tear factor at 300 ml. freeness
TAPPI burst factor at 300 ml. freeness
Normal prodn. cooks—75 min. at 100 p.s.i.—4300 lb. AA (6600–6800 total chemical)—20–24% sulf.—chips not pkd.
Avg.= 22.3
Avg.= 3.7
Avg = 128
Avg.= 73
Trial cooks of Nov., 1958–75 min. at 100 p.s.i.—8000 lb. total chemical—27–30% sulf.—chips packed
Avg. = 19.7
Avg = 2.8
Avg.= 126
Avg. = 75.9
Trial cooks of Aug.–Sept., 1959–65 min. at 100 p.s.i.—7200 lb. total chemical 27–30% Sulf.—chips packed
Avg.= 21
Avg.= 2.2
Avg.= 131
Avg.= 75
NUMBER OF TESTS

Fig. 8 Histograms of pulp properties from digester runs

The results of this experimental run together with those for normal pulp production are presented in Fig. 8. All tests refer to pulp from "B" digesters.

On comparing the experimental run (November, 1958) with normal production results, it is evident that the experimental cooks did not achieve entirely the results anticipated. Pulp permanganate number was lower and burst strength was higher as predicted; however, there was a slight decrease in tear strength. Per cent rejects were slightly higher than predicted. Based on this and the knowledge that nominal chip size had increased during the run, the results were deemed inconclusive and the next run was scheduled. This next experimental run was designed to:

1. Produce permanganate numbers slightly higher than the first run.
2. Improve pulp strength properties.

The run took place in August–September, 1959, and consisted of 5 days' operation of all eight digesters. For purposes of comparison, the trial conditions for "B" digesters are listed:

White liquor sulfidity, %	27–30 (by adding S at digesters)
Total chemical per cook, lb.	7200
Total liquor volume, gal.	7900
Chips per cook, lb.	71,000—packed—50% moisture
Total chemical, %	20.3
Cooking time at 100 p.s.i., min.	65
Type wood used	Southern pine; chips less than 30 days old.
Nominal chip size	$^1/_2$–$^3/_4$ in.

As shown at the bottom of Fig. 8 this run was entirely successful. Not only were per cent rejects considerably below normal, but pulp strength tests were significantly increased. In addition, digester production capability was increased 15%.

CONCLUSIONS

As a result of the commercial pulping trials at Gulf States Paper Corporation's Tuscaloosa plant, the following trends were established:

1. Production of relatively soft cooks in the kraft process does not necessarily decrease strength properties.

2. Southern pine cooks utilizing relatively high total chemical (20–22.5%) and high sulfur content in white liquor tend to maximize pulp strength. This also produces lower per cent rejects. Optimum results were obtained at 21 average permanganate number with nominal chip size of $^1/_2$–$^3/_4$ in. Sulfur was added at the digesters to create an equivalent sulfidity of 27–30%.

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Evaluation of Straw Pulp and Hardwood Pulp Mixtures

GEORGE VÁMOS AND TIBOR MÉRO

The extensive use of straw pulp causes some difficulties on the paper machine (inadequate drainage, picking on the press, etc.). The increasing amounts of hardwood pulps (poplar) in the paper furnishes—in view of the softwood shortage—may result in the elimination of such difficulties caused by straw pulp. To determine the properties of straw pulp and hardwood pulp mixtures special care is given to the study of the dynamic properties (tear, double fold), which are mainly responsible for the unsatisfactory processing of paper. The drainage properties of the various pulp mixtures are also thoroughly discussed. The effect of the addition of softwood chemical and mechanical pulp on the different properties of the mixtures is described.

THE NOTABLE and steady development of the paper industry has, even in technically and culturally advanced countries, raised serious problems of raw material supply. Conifers, the chief raw materials for papermaking for a long period, are for climatic, topographic and other reasons not available at all or are not in sufficient quantity in most countries. In fact, it is only the northern countries that are free from troubles of timber supply, even though in some of them the distances from sources to mills also create difficulties.

Besides conifers, the raw materials suitable for papermaking are almost exclusively short fibered, with an average fiber length of about 1 mm.

Obviously, in their own interest, all countries have to exploit to the greatest possible extent their existing sources of raw material—partly available as waste products—and this means, in our case, that we have to examine thoroughly for utilization in the papermaking industry all suitable hardwoods, annual and perennial plants. For the further expansion of the paper industry this is exceptionally important in most countries of western, central, eastern, and southern Europe, as well as in a number of overseas countries.

It is known that several peculiarities of the short-fibered raw materials lower the quality of the paper to be produced and, moreover, difficulties arise during the manufacturing process. In this study we deal with the examination of some characteristic properties of raw materials, on the basis of the research work carried out at the Hungarian Paper Research Institute. The aim of this research work was to produce mixtures—by using short-fibered materials—satisfactory both for the manufacturing process and for their papermaking characteristics.

The pulps made of broadleaf trees (poplar, birch, beech) just as those made of annual plants (various straws) differ in many respects from the coniferous pulps for papermaking-fiber dimensions, fiber-morphology and, because of these factors, also in their behavior on the paper machine and, last but not least, in the properties of the paper sheet itself.

If these raw materials are to be used in large quantities, it is not only their characteristic properties that must be thoroughly known, but they have to be applied in mixtures in which the inferior properties of one material are counterbalanced by the advantages of the other component. Moreover, the machinery has to be correspondingly adjusted to eliminate difficulties in processing.

It should be borne in mind that the advent of groundwood, which had so essentially differed from the rag pulp hitherto exclusively used for papermaking, caused great trouble to the papermakers of a 100 years ago. On the other hand, the application of groundwood and chemical pulp coincided with the era of mechanization and so the constructional development of the paper machines was in accord with the requirements of the different new raw materials.

However, this is not the case with the recently expanding use of short-fibered materials. The paper industry has now to apply these on their paper machines which had been developed for maximum capacity and constructed primarily for the use of coniferous chemical woodpulp and groundwood. Technological difficulties are met, in the first place, on the wet end of the machine, in the course of sheet formation and pressing, because these short-fibered pulps present troubles, chiefly from their more difficult drainage, lower wet web strength, and tendency to pick at the press rolls. Further difficulties are connected with certain qualities of the paper—primarily with its dynamic strength properties. Therefore, further on in the process, breaks may occur between the presses and the driers, or in the drier part itself. Finally, these defects may appear also in finishing the paper or during use.

While the first group of difficulties is relatively easy to combat—by the insertion of a longer wire, by increasing in the suction areas, by using lower vacuum—the behavior of the paper in finishing and its better and wider application can be influenced by the proper selection of raw materials and their appropriate refining, leaving aside the treatment with different synthetic and other materials during or after the beating process.

All these findings were based on prolonged laboratory tests combined with mill experiences, which necessitated further studies in this direction to clear these phenomenons, eliminate these difficulties. Here it should be pointed out that without careful examination apparent contradiction may be discovered between the laboratory results and those gained in the mill. In fact, many short-fibered pulps possess excellent static strength characteristics that will class them as suitable—at first appearances. From our mill practice, however, it was clear to us that a judgment must not be based on these properties only, but the dynamic strength, and other characteristics must be taken into consideration, e.g., those causing picking at the rolls. The fall in dynamic strength characteristics greatly increases the risk of breaks in the web on the wet end, also between the driers and, in extreme cases, it may cause such an amount of broke, that work on the paper machine becomes almost impossible, even if the picking of such short-fibered materials as rice straw pulp could be successfully overcome.

EXPERIMENTAL PROCEDURE AND RESULTS

Further studies, as a continuation of our earlier work (*1–3*) were carried out with the aim of determining the proper ratio of short-fibered raw materials in 2, 3, and 4-variable pulp mixtures.

The investigations were carried out on three different bleached pulps, the main characteristics of which are shown in Table I. These materials were separately beaten in a Jokro mill to various freeness, then mixed—as described later with mechanical pulp also—and made into sheets at 100 g. per sq. m. substance on a Rapid-Köthen sheet-mold.

Table I. Some Main Characteristics of the Investigated Pulps

	Sulfite spruce pulp	*Sulfate poplar pulp*	*Sulfate wheat straw pulp*
Alpha-cellulose, %	85.0	83.5	76.2
Pentosans, %	5.9	20.0	23.8
Lignin, %	0.3	0.9	1.9
Ash, %	0.34	0.69	1.15
Brightness, %	82	77	80

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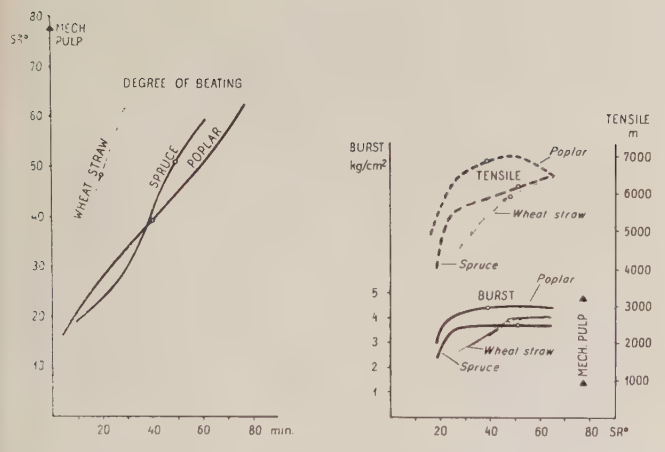

Fig. 1. Pulp characteristics

At the same time the drainage characteristics of the sheets, and after corresponding conditioning (20°C., 65% R.H.) the main physical characteristics, especially the dynamic strength values were determined. The dynamic strength properties strongly affect the manufacture as well as the application.

Our chief aim has been to get acquainted with the behavior of the various mixtures on the wire and on the press rolls. Therefore, the drainage was investigated by a special device and the picking tendency with the aid of wet strips on an apparatus developed earlier (*1*). Both testing methods are described in the following evaluations.

The main physical characteristics of the investigated chemical and mechanical pulps are shown in Figs. 1 and 2.*

It will be seen that the materials differ considerably in some of their characteristics so that, based on our earlier work, certain promising results may be expected from their blending.

The following observations were made for the materials tested: the freeness of spruce and poplar pulp develop on fairly similar lines, while the freeness of wheat straw pulp decreases more rapidly with beating.

At lower freenesses the drainage time of poplar pulp differs from that of spruce pulp, whereas the drainage time of wheat straw pulp shows higher value all through the beating process.

Burst and tensile strength values differ very little for the three materials, though they decrease in the following order: poplar, spruce, wheat straw. The order for double fold and tearing strength puts spruce pulp first and wheat straw pulp gives low double-fold values in the materials tested.

* We regret that at the time of our investigations we were still not familiar with the method of measuring the water retention value (W.R.V.) introduced by Jayme and Krüger (*7*), nor with the method of wet porosity showed by Imset (*8*).

These and perhaps some other new methods still, surely will help in future investigations to get comparable values which are convenient for the practice too.

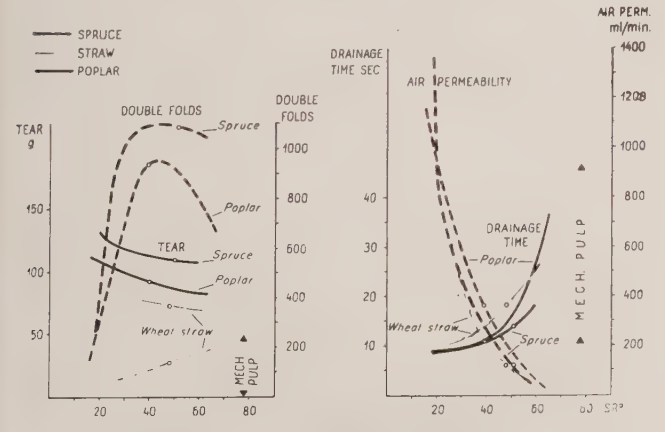

Fig. 2. Pulp characteristics

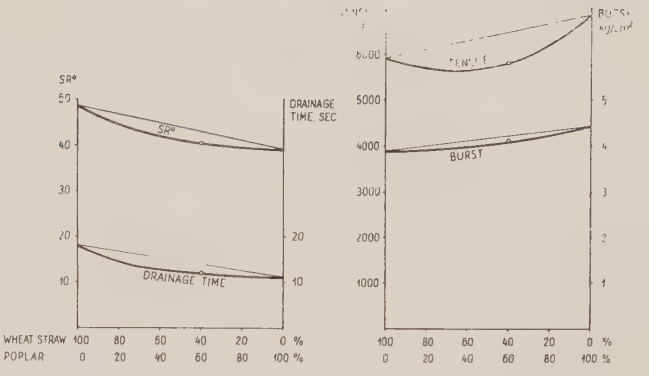

Fig. 3. Characteristics of wheat straw pulp and poplar pulp mixtures

For the tests the different materials were beaten separately and blended in different proportions. The beating times were chosen so as to give optimum properties.

Poplar pulp was beaten for 40 min. (39°S.R.) because both drainage time and tearing strength are still very good at this stage, while static strength characteristics and double fold have already developed. For spruce sulfite pulp, a beating time of 50 min. was chosen (51°S.R.) to obtain the best possible strength values. A satisfactory tearing strength makes a slight decrease of static strength properties acceptable. Straw pulp was beaten for 20 min. (48°S.R.), as tearing strength and burst is good at this stage, but when beating further, drainage time is increasing rapidly.

The analysis of our investigations raises interesting problems. For example, in spite of its long drainage time and its unfavorable dynamic strength properties, groundwood pulp can be satisfactorily processed on the paper machine. This fact raises the question that, if paper can be made from, say, a mixture of 80% groundwood and 20% sulfite pulp even at highest speeds on the paper machine, why do the familiar difficulties arise with certain short-fibered pulps—even if at lower percentages in the mixture? This problem seems to be very important and should form the central point of future investigations.

Returning to our recent researches, let us now examine the mixtures with changes of composition in steps of 20%.

Wheat Straw Pulp and Poplar Pulps (Beating Time, 20 and 40 Min., Respectively)

As can be seen from Figs. 3 and 4 drainage and static strength properties of the two materials are not substantially different, but dynamic strength properties of poplar pulp exceed those of wheat straw pulp. The freeness and drainage time of the mixtures are better than the values derived proportionately from the values of each of the components, whereas the strength characteristics—contrary to the results of our previous investigations on spruce and straw pulp—are slightly worse than those calculated, in the same way. This is obviously due to the fact that we have dealt in this case with a mixture of two short-fibered materials in which

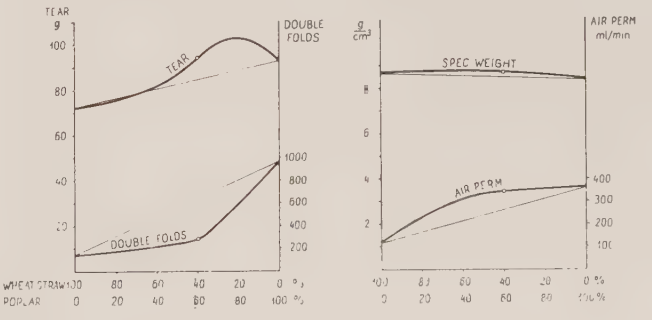

Fig. 4. Characteristics of wheat straw pulp and poplar pulp mixtures

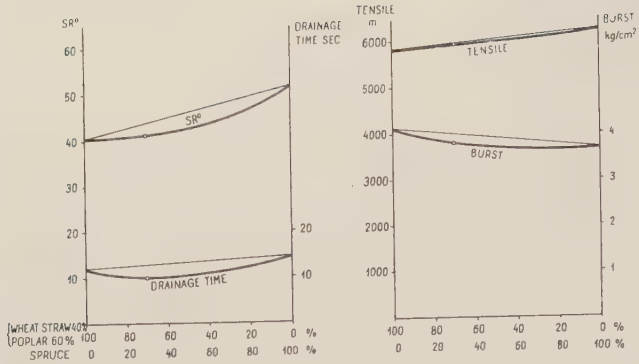

Fig. 5. Characteristics of wheat straw pulp (40%), poplar pulp (60%), and spruce pulp mixtures

bondings, etc., develop differently than when one of the components is spruce pulp.

As our main object is the utilization of nearly equal amounts of wheat straw and poplar pulps, we chose for our further tests a mixture of 60% poplar and 40% wheat straw pulp. With this mixture we obtained good drainage, a fairly good tearing strength; only the double fold was not quite satisfactory. The static strength properties were good too and the air permeability was very good. A further increase of the proportion of poplar pulp to improve double fold, however, would lower undesirably the proportion of wheat straw pulp in the mixture. Therefore, we assume that by further blending with spruce pulp, the double-fold characteristics would improve to a certain extent—as will actually be proved by subsequent tests.

Mixture Containing 40% Wheat Straw and 60% Poplar with Spruce Pulps (Beating Time 20, 40, and 50 Min., Respectively)

As shown in Figs. 5 and 6 the freeness and drainage time—similar to the preceding tests—have turned out to be better than the proportionally calculated values of the components. The static strength characteristics and air permeability, too, turn out better in these mixtures, whereas the bulk does not show any interesting changes whatever.

On the basis of the previous experiments, for subsequent examinations of mixtures with mechanical pulp, we have chosen a mixture containing 30% spruce pulp, a 28% wheat straw pulp, and 42% poplar pulp.

Although the dynamic strength properties of this mixture are somewhat below those of the spruce pulp, yet, compared with the 100% wheat straw pulp and with 100% poplar pulp, respectively, from the point of view of utilization, some rather favorable results have been reached as shown in Table II.

Various Mixtures of Chemical and Mechanical Pulps

In different mixtures of the above (30% spruce, 28% wheat straw, and 42% poplar pulps) and mechanical pulp, the resulting chief characteristics are shown in Figs. 7 and 8.

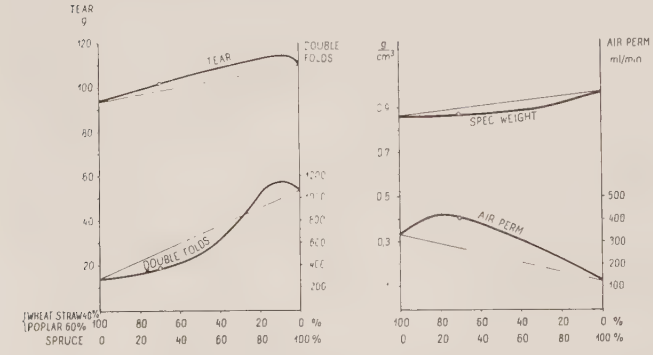

Fig. 6. Characteristics of wheat straw pulp (40%), poplar pulp (60%) and spruce pulp mixtures

Table II. Physical Characteristics Obtained with the Suggested Mixture

	Mixture of 30% spruce, 28% wheat straw, and 42% poplar pulp	*100% Wheat straw pulp*	*100% Poplar pulp*
Freeness, °S.R.	41	48	39
Drainage time, sec.	10	18	11
Tensile strength, m.	5950	5900	6900
Bursting strength, kg./sq. cm.	3.8	3.9	4.45
Tear factor	101	72	92
Double fold	380	140	940
Air permeability, cc./min.	410	120	360
Specific weight, g./cc.	0.87	0.86	0.84

Thus, the addition of about 20 to 25% mechanical pulp (we took a mechanical pulp of about 75% spruce and 25% poplar) causes hardly any change in the static strength properties, only the double fold drops considerably, even beyond its proportional value, while there is only a slight reduction in the tear factor. With this mixture there is hardly any rise in the drainage time, and even some improvement may be experienced in air permeability. It may thus be concluded that, by the addition of small quantities of mechanical pulp, no considerable divergence in the important characteristics occurs.

The comparison of the pulp mixture in question with a mixture of pure spruce pulp and 75% groundwood was of considerable interest. Its result is also shown in Figs. 7 and 8.

The investigations have shown that the blend of the said chemical pulps and 75% groundwood, as well as that made of pure spruce pulp and 75% groundwood, revealed hardly any difference in the properties tested—the values obtained were almost identical. This would mean that, in practice, provided no other circumstances prevent processing on the paper machine, such mixtures may be used to advantage for substitution of pure spruce pulp!

These theories are supported by mill experience gained in Hungary and also in other countries. Such experience has proved that no substantial difficulties have been encountered with the employment of pulps blended with relatively small quantities of short-fibered pulps.

Behavior of Mixtures in the Wet State

The above statements do not contain any data on the behavior of the tested mixtures in the wet end of the paper machine. In one of our earlier papers (*1*) it had been pointed out that the behavior of materials on the paper

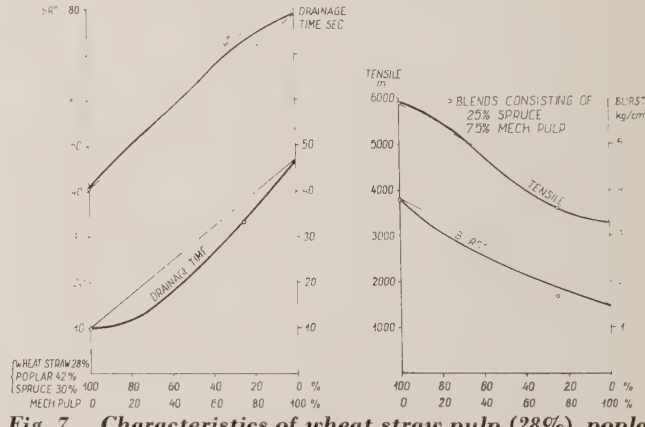

Fig. 7. Characteristics of wheat straw pulp (28%), poplar pulp (42%), spruce pulp (30%), and mechanical pulp mixtures

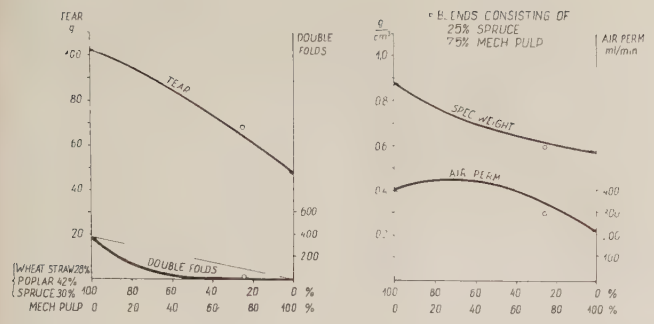

Fig. 8. Characteristics of wheat straw pulp (28%), poplar pulp (42%), spruce pulp (30%), and mechanical pulp mixtures

machine is affected by various factors such as wet strength, dry solid content ahead of the first press roll, tendency to pick up to the press, etc. Several publications have appeared lately on the drainage of materials on the wire at different vacua and for different lengths of time (*4–6*). For examining the drainage of the mixtures on the wet end of the machine, similar equipment was used. The sheets drained on the Rapid-Köthen sheet machine without vacuum were subsequently subjected to vacuum in the test equipment for different periods (Fig. 9). Suction time varied in the range of 0.1 to 5 sec. Vacuum values can be varied widely, depending on the air permeability of the sheet, the performance of the suction pump, and the capacity of the suction tank. In our investigations the suction pump used had a slight discharge of 375 l. per min. at 0°C. and 180 mm. Hg vacuum, and the connected suction tank had a volume of 1000 l. The experiments were made only to the extent that no noticeable loss of vacuum resulted.

Vacua ranged within 15 to 35 cm. mercury, but comparisons were based on the 18-cm. value.

The dry content of different mixtures determined by applying suction for 0.1 to 1 sec are shown in Fig. 10. As can be seen, the dry content decreases while the freeness and the drainage time drops, although the values are not in direct correlation. The drainage characteristics of different pulps, although showing similar trends take different courses, depending on their structure and swelling properties. Thus, for instance, it seems obvious that by using a suction time of 0.3 sec., a poplar pulp of 40°S.R. reaches 16.8% dry solids content, while wheat straw pulp of 38°S.R. only 13.4%; but it is difficult to explain that at identical conditions of vacuum and suction time, spruce chemical pulp of 51°S.R. attains a dry solids content of only 9.8% and a 78°S.R. mechanical pulp one of 7.9%. It is well known that proper operation cannot be expected on the paper machine below a dry solids content of 16% after the couch. Of course, in practice the dry solids content can be raised by using a correspondingly lower vacuum, to ensure trouble-free leading of the paper web through the machine.

We have also tested the tendency of picking at the press rolls of various pulps (*1*). For this purpose three wet strips, each 30 by 170 mm., were taken from the sheet machine with the aid of wire frames. These, with the aid of felts,

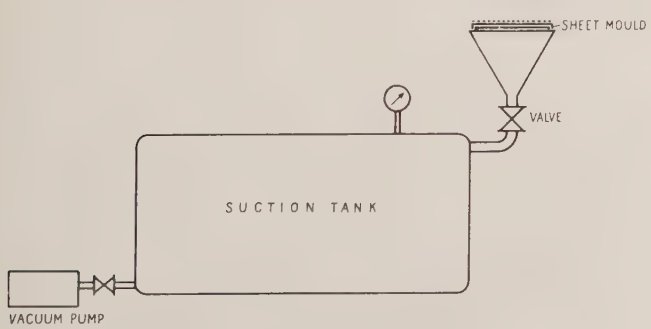

Fig. 9. Suction device

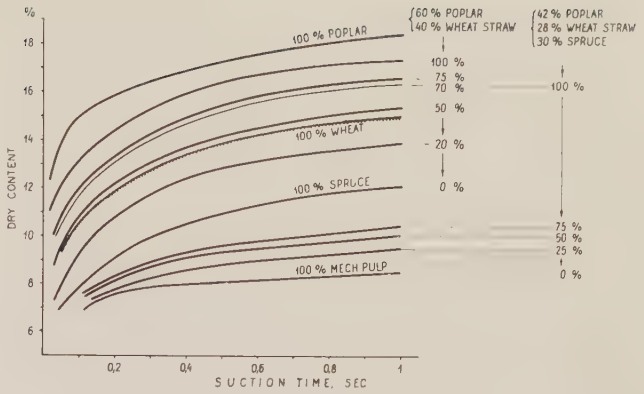

Fig. 10. Dry content of different pulp mixtures at various suction times

were then pressed against the circumference of a metal cylinder, at a pressure of 4 kg. per cm. Subsequently, according to a method developed in the course of earlier experiments, the work needed for peeling off the strips was measured. It has been found that this work decreases almost linearly with the increasing dry solids content, prior to pressing, and it reaches its minimum with all kind of pulps at about 23% dry solids content (Fig. 11). The work needed to peel off the strips naturally varies with different pulps, but it shows a maximum at about 12% dry solids content with the investigated blends. At a lower dry solids content the pulps pick at the roll to such an extent that they cannot be peeled off without a break. Examining the tendency to pick, the spruce chemical pulp tested stands first, followed by the poplar and wheat straw pulp. The fact that the dry solids content of spruce pulp can be reduced to 12%—in spite of its greater picking—can be attributed primarily to its relatively higher wet tensile strength. Considering, on the one hand, the peeling-off work at a stage when this causes the web to break, and examining the wet tensile strength of the pulps on the other, we arrive at the values shown in Table III. That means that the variations in wet web strength and in the peeling-off work with different pulps, show the same trend.

However, the behavior of mechanical pulp considerably differs from that of chemical pulp, as in spite of the significantly lower wet web strength during the tests, no picking up that caused a break has been experienced. For instance, mechanical pulp with even as low a dry solid content as 9 to 10% may be successfully peeled off. Although the work involved in peeling-off is comparatively high (75 g.-cm. at 9–10%, and 55 g.-cm. at 15%) the wet tensile strength (160–210 g.) is much below that of the chemical pulps (370–500 g.). This indicates the influence of some other factors, so far insufficiently clear.

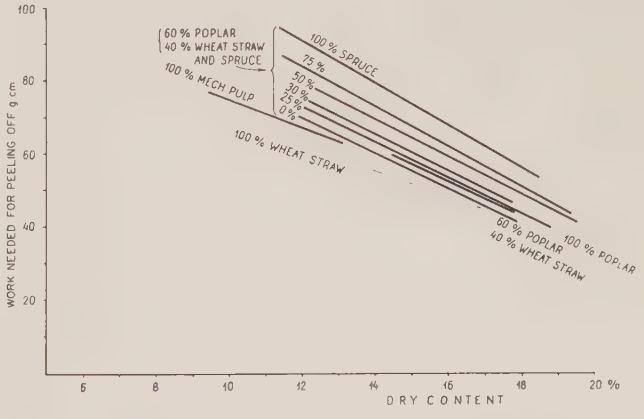

Fig. 11. Peeling-off work of different pulp mixtures at various dry contents

Table III. Characteristics Influencing the Wet Web Adherence

	Work needed for peeling off, g.-cm	*Wet tensile strength, g.*
Spruce chemical pulp	96	500
Poplar pulp	72	390
Wheat straw pulp	65	370

Important and interesting work for the future would be the investigation of these properties of mechanical pulp.

This is confirmed also by Fig. 12, showing the characteristics of the mixtures of three chemical pulps and mechanical pulp. It can be seen that the dry solid content may be diminished by increased admixture of mechanical pulp without the risk of a break and, at the same time, the higher chemical pulp content increases the wet strength and the peeling-off work is thereby raised without the risk of any paper adhering.

Examining this property, we arrive at the interesting conclusion that the addition of even a relatively small quantity of mechanical pulp favors safe peeling of the web of the press roll, which is very important for the behavior of the paper on the machine, and definitely imparts groundwood characteristics to the paper being made.

These tests represent, of course merely a part of the extensive work aimed at accessing the suitability of short-fibered materials on the paper machine. The results achieved so far have proved that short-fibered pulps of suitable quality and composition can be used to advantage. This fact furnishes a basis for the proper admixture of blends and, at the same time, encourages the papermaking industry to a more daring utilization of short-fibered pulps, which many countries are ever more compelled to resort to, lacking sufficient raw material supplies.

SUMMARY

Mixtures containing spruce pulp and short-fibered materials (poplar and wheat straw pulp) in different proportions, were tested with and without an admixture of spruce and poplar mechanical pulp. The tests aimed at finding a mixture free of the undesirable behavior of short-fibered stocks: slow drainage on the machine, tendency to pick at the press rolls, breaks between the driers on the paper machine and other disadvantageous properties. With this aim in view, the chief characteristics of a suitable mixture have been determined—its drainage, strength properties and dry solids content as a function of the vacuum applied.

The investigations have shown that while dry solids content decreases in the order poplar, wheat straw, spruce, and groundwood, the work required for peeling the web off the press is highest with spruce pulp and is followed by poplar, wheat straw, and finally by groundwood pulp. This means that the work of peeling, apart from the moisture content of the mixtures, follows from their structural features, and its maximum is determined by wet tensile strength.

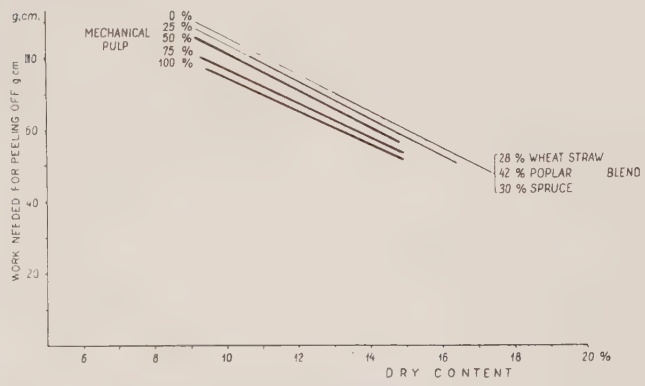

Fig. 12. Peeling-off work of different pulp mixtures at various dry contents

The tests have proved that a suitable and proper blending ratio enables the utilization of more short-fibered raw materials. Mixtures containing nearly equal quantities of wheat straw and poplar pulp and about 30% of spruce pulp, promise good results.

The admixture of groundwood (up to 20 to 30%) does not considerably affect the characteristic values of the mixtures.

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Case Sealing at the Camas Converting Plant

RICHARD L. CARLSON

In November 1957 production of the Camas converting plant had increased to the point where the case sealers were not capable of handling the volume. Over 100,000 cases were produced per month in 70 different grades and 15 different case sizes. The first improvement was to increase the speed of the sealers from 6 to 12 cases per minute. It was first thought heat would be required on the case flaps to insure sealing at this increased speed. However it was found the cases sealed satisfactorily with no heat. The second improvement was to rearrange the sealers for better material flow. It was found 58% of the cases were all the same size so an automatic case selector was devised to select these cases for automatic sealing. The results of these improvements were improved working conditions, increased capacity of 80%, reduced case damage and tangible savings of $18,000 per year.

In November 1957 the converting plant superintendent was aware the case sealing arrangement was no longer adequate to handle the continually increasing production of his department. At that time he asked the Industrial Engineering department to study the case sealing at the converting plant to determine how the capacity of the sealing installation could be increased and sealing costs reduced. The benefits that were to be expected from this study would be: (1) increased capacity; (2) improved working conditions; (3) reduction in case sealing costs, and (4) reduction in case damage at the sealers.

At the time this project was started the converting plant was producing over 100,000 cases per month. This consisted of 70 different products. These 70 products included colors, assorted and half size cases of the same basic product and were packed in 15 different sized, corrugated cases. The number of different products that could be produced at any one time was 25. All of these cases were being sealed with three case sealers located on the coverting plant mezzanine.

The rate the cases come to the sealers is not constant but varies with the assorting of colors in the converting plant.

The following is a description of the case sealer layout, as

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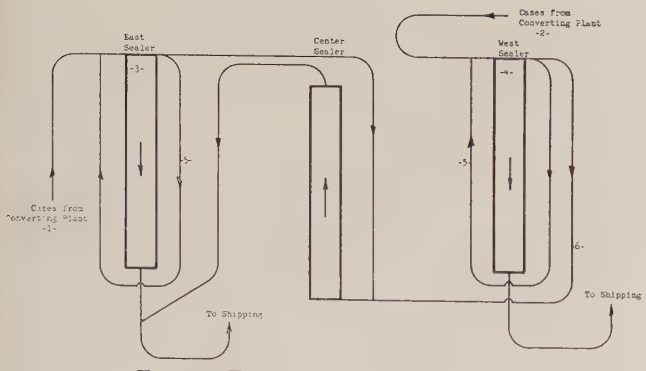

Fig. 1. Existing case sealer layout

it was when the study was conducted. Temporarily the layout is still the same. The parts for the improvements described later in this paper have been purchased and delivered and are now awaiting installation.

Figure 1 is a flow diagram of the present layout. The numbers referred to are shown in Fig. 1:

1. The unsealed cases come up from the converting plant machine room floor on two powered belt conveyors (1 and 2).

2. The sealerman (3 and 4) can put the case in the sealer, on the runaround (5), divert it to the center sealer or, in the case of the east sealerman, manually stack off one odd size case. Only one size case can be sealed at a time, therefore the runarounds were provided for temporary storage of cases not of the correct size for sealing. The center sealer was not provided with a runaround; therefore only one size case from each sealerman can be diverted to it at a time without having to stack out.

3. In addition to cases coming to the west sealerman, boxes of cull rolls and broke are also sent to him. This broke is put on the conveyor to the center sealer and is handled from there by a waste handler who also runs the center sealer when necessary.

4. After being sealed the cases are conveyed to the shipping department on the next higher floor.

With this arrangement it is possible for a case to be handled five to seven times before it is sealed. The sealer size changes are made by hand. The location of the cranks to change size are not in a convenient location to the operator's working position and require considerable time and effort to operate.

Some cases are damaged by the operator putting two of the smaller cases in one flight of the gluer. This is possible because the operator is able to put the cases directly into the gluer. The combined weight and friction of the two cases is enough for the pusher bar to crush the last case.

Two distinctly different approaches were used to find a solution to this problem. The first was to determine if the present sealers could be operated faster in the existing location. The second was to attempt to relayout the sealer area for better flow.

A speed-up of the sealers required that the existing press sections be used. There was no room for an additional length of press to be installed. The lengths of the three press sections were all 19 ft. long and the gluers were running at 5 to 6 cases per min.

The speed of one of the sealers was increased to 12 cases per min. and three trials run on it. The first was to use a fast setting resin-base glue. This glue machined and sealed very well at the increased speed. In fact it appeared it would have sealed running much faster. On the basis of a very short run with this glue, the glue cost per case was six times the cost of our own glue. This, it was estimated, could be reduced by one half by over hauling of our sealer which on one glue pot could not doctor the glue thin enough. Even at this reduced cost it could not compete with the dextrine type glue we make ourselves.

This same resin glue is used very satisfactorily in other Crown Zellerbach mills where the conditions and requirements are different.

The second trial was to run our own glue with heat applied to the case flaps before the glue was applied and also to heat the glue in the glue pots. Five-hundred watt strip heaters were added to the formers and hold-down plate of the gluer and immersion heaters were added to the glue pots. The cases sealed satisfactorily except for the heated glue pots which will be explained later. The third trial consisted of reducing the heat on the gluer. It was found the cases sealed satisfactorily with no heat at all.

No further increases in sealer speeds were tried because it was felt the maintenance problems would offset any gain in increased speed.

The total cost of the changes necessary to speed up the sealer was about $125 for material and $250 for labor.

The strip heaters on the formers worked very well and probably would allow a speed increase on any sealer arrangement. The heaters in the glue pots were a different story however. The increased heat of the glue evaporated the water so rapidly the operator was continually having to add water. The amount of water required was estimated by the operator and he would add more than necessary in an attempt to reduce the number of times he had to add water. This additional water required a longer time for the glue to strike in. The only apparent solution was to cover the glue pots and on our machines this was not possible without making it difficult for the operator to add glue to the pots as necessary. Heating of the glue did not seem to be a satisfactory solution.

It was felt the sealerman could handle the cases about 50% faster with this increase in speed. More wasn't possible because of the way the cases came to him and the handling required.

The second area of possible improvement was in a re-layout of the sealing area, or new equipment, or both.

Any expansion of the sealer area would have to be into room now used by the shipping department and at a premium, suspended overhead in the converting plant which would be a maintenance problem and a safety hazard, or extend the mezzanine floor which would be expensive.

A case segregating layout where pallet load quantities of each product being produced, would be accumulated on separate storage conveyers and then sealed had been previously investigated and was now reviewed. The cost of such an installation was approximately $60,000. This was not attractive because of a low return on the investment from a savings standpoint, and also because of a poor utilization of warehouse space required by the installation. With this type of installation the mezzanine, where the sealers are now installed would not be a useful area because of limited access.

In looking at the products from the converting plant it was found that 58% of the cases were all the same width and height. This meant that if these cases could automatically be selected by size they could also be automatically sealed.

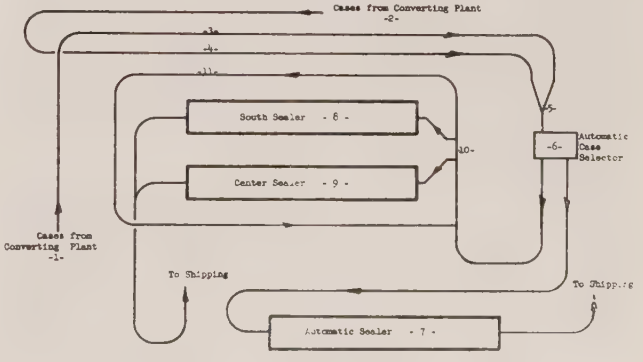

Fig. 2. Proposed case sealer layout

Mathews Conveyor Co. said they could build such a unit so the sealers were relocated using this principle.

The following is a description of the future case sealer layout. Figure 2 is a flow diagram of this system. The numbers referred to are shown on Fig. 2:

1. The unsealed cases come from the machine room floor in the same manner as in the present layout (1 and 2).

2. The cases are accumulated on two conveyors (3 and 4). One of these will always be running and possibly both.

3. The two incoming lines of cases are converged (5) into one line and the cases then go into the automatic case selector. The operation of this selector is: (a) All the cases are forced to one side of the unit. (b) One of the lengthwise flaps is plowed into a vertical position. (c) Two microswitches in the floor of the unit detect the correct width of the case; if neither switch is actuated it is not the correct width, if both switches are actuated it is not the correct width, if one of the switches is actuated and not the other it is the correct width. (d) Two photoelectric eyes are installed in the sides of the unit to detect the correct height. The same method of operation and detection applies to the electric eyes as it did to the microswitches. If one eye is actuated and one is not, then the case is the correct height. (e) If the case is the correct height and width a group of chains and pusher bars moves the case sideways to an adjacent parallel conveyor.

4. The cases that have been selected to be automatically sealed are conveyed to an automatic sealer (7) and then to the shipping department. This sealer is a new one for this department, having been used in the napkin department and now no longer required there. The speed of this sealer is to be 17 cases per min.

5. The remaining cases are conveyed to the two other sealers (8 and 9). The operator (10) can put the cases into either of the two sealers or onto the case runaround (11). These two sealers will have powered size changing drives installed, these will enable the operator to change the sealer size without leaving his station. The sealed cases are conveyed to the shipping department as in the present system.

The principle of detection of this selector is apparently new and is felt to be a contribution to case selection where weight of the cases cannot be used or coded eye spots on the sides of the cases are not acceptable from an advertising viewpoint.

With this arrangement all three of the sealers are located in a position to be used at all times. The number of handlings per case has been reduced considerably by providing the powered size changing on the two hand fed sealers, the operator will change sizes more frequently and will not accumulate as many cases of one size before he seals them.

Case damage will be reduced by not storing on the runaround corners, as is done now, and also by providing a short length of approach conveyor to the sealer. This conveyor will prevent the sealerman from putting two cases in one flight of the gluer.

The total cost of this rearrangement is $22,000 and, as a result of this change, a labor savings of $18,000 per year will be realized. The savings are based on reducing the crew by one man on the first and second shifts and by releasing the waste handler from having to operate a sealer for part of his shift, thus allowing him to be scheduled for work presently done by part-time people.

The four benefits or goals of this project were all reached in varying degrees:

1. The capacity was increased approximately 80%.

2. The working conditions were improved by reducing the number of cases the operator had to handle and rehandle.

3. The case sealing costs were reduced by a tangible $18,000 per year.

4. Case damage was reduced to a lesser degree by improved layout.

These achievements were all made possible through the application of work simplification principles of looking at the parts of the problem and questioning the reasons behind the methods.

A Shibley Award paper presented at the meeting of the Pacific Section of TAPPI, held in Camas, Wash., April 7, 1959.

A Laboratory Instrument for Measuring the Piling Propensity of Papers Used in Offset Lithography

FRANK F. OEHME and DONALD C. NEPPER

A definition for piling is given as it pertains to coated lithographic grades of paper with a distinction being made between piling, tinting, wet pick, and wet rub. The conditions which are necessary to produce piling in multicolor offset printing are discussed in relation to the problem of reproducing these conditions in the laboratory for establishing a control test for measuring this property. A detailed description of the development, construction, and operation of an instrument that measures the relative potential of an offset sheet to induce piling is presented as a measure of that sheet's resistance to wet pick.

PILING is the accumulation of ink on rollers, plates, or blankets when one of the transmitting surfaces retains more ink then it transfers.

The problem has developed in direct proportion to the increased number of large four- and five-color offset presses being used in the lithographic industry. This is because piling resulting from coating particle contamination is usually peculiar to multicolor presses where the effect of water applied to a coated paper surface on the first unit can result in a piling of ink on succeeding blankets. As Beckman (*1*) points out, piling is only one of a set of problems aggravated by adverse interactions between paper, ink, and fountain solutions. Beckman describes piling as occurring only on the image areas of a blanket, while Reed (*2*) also includes the buildup of ink in the nonimage areas of a blanket.

In the Printing Ink Handbook (*3*) reference is made to piling as the accumulation of ink on a blanket due to the destruction of the pigment carrying properties of the vehicle. Several examples are cited explaining how the pigment-to-vehicle balance can be adversely affected by fountain solutions, moisture, high humidity, and paper dust or coating. Paper can influence the pigment carrying power of a vehicle by:

1. The introduction of minute particles of coating *into* the vehicle.

2. The introduction of moisture *to* the vehicle which may or may not be absorbed or repelled.

3. The selective absorption of vehicle from the ink.

The paper properties involved in these theories are wet pick resistance, wet rub resistance, surface smoothness, porosity, and water absorbency.

We think the wet pick resistance property of paper is the most important one to control in the reduction of piling as influenced by paper under normal lithographic process conditions. The other properties of wet rub resistance, smoothness, porosity, and water absorbency, are not causes in themselves but are dependent upon the exposure to *excessive* moisture conditions.

These conditions of high humidity, and excessive press moisture are abnormal and more closely related to the problems of nonimage area piling and tinting than they are to image area piling which we have primarily concerned ourselves with.

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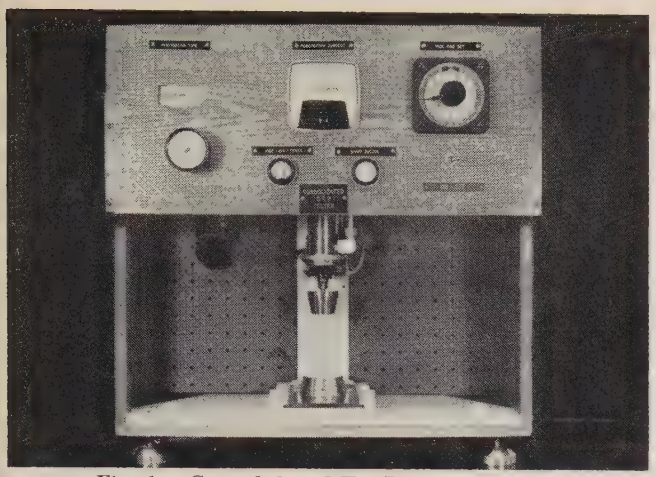

Fig. 1. Consolidated Tri-Pick instrument

This report deals only with point no. 1 or the way coated papers, particularly with casein or protein adhesive, can induce the piling of ink on blankets of multicolor lithographic presses. It is confined primarily to that piling which is observed only in the image areas of a blanket, generally as a result of what might be referred to as wet picking of the coated surface with associated ink contamination and buildup.

SCOPE

The basic problem was to develop a simple laboratory test that would show from one sheet, the cumulative effect of thousands of sheets as they are exposed to the forces applied during four-color wet printing on offset presses. It became necessary to reproduce piling by creating it on a two-color press studying the forces involved and duplicating those forces in a controlled concentrated way in an instrument.

Extensive experiments were conducted with existing tests such as wet rub, the "wet thumb test," and wet and dry IGT tests to evaluate their application to the problem. Correlation analyses were made to the results obtained from running fourteen papers from various manufacturers on a Harris two-color press using the following technique.

A clean de-sensitized plate was used on the first unit to apply water to the sheets.

A clear lithographic overprint varnish was printed on the second unit with a specially designed form containing solids, reverses, and halftones in a layout sensitive to piling effects. Any residue of coating material accumulating on the blanket was easily recognized through the varnish.

Each sample was run at the same speed and ink and water control settings for each test.

Scotch tape replicas of the blanket were obtained after 100, 300, 500, 1000 and 2000 impressions from each sample.

These were ranked according to the quantity of coating material apparent on the tapes and the process repeated at a different speed or water control adjustment.

Fig. 2. Operation of consolidated Tri-Pick instrument

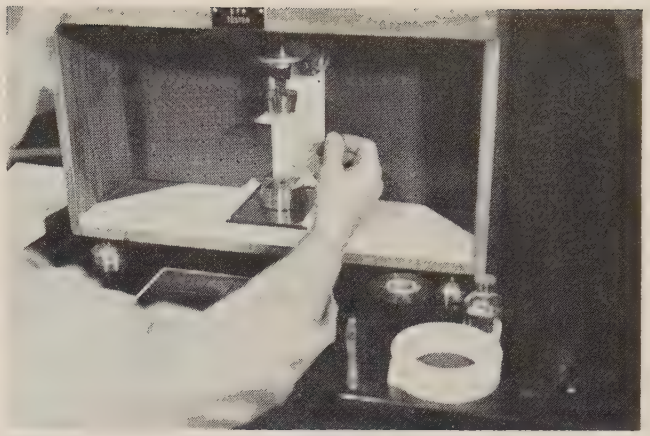

Fig. 3. Applying fountain solution

The empirical data thus obtained was used to judge the value of all the other experiments conducted with other testing methods. It is interesting that the "wet thumb pick test" described by Reed (*2*) proved to be the most significant. It is also interesting to note that papers with low wet rub ratings did not necessarily pile, while some of the samples with excellent wet rub resistances did pile. Apparently this is because the type of contaminant contributing to ink piling is mostly water insoluble but oil miscible and disturbs the pigment carrying property of the vehicle. Papers, with poor wet rub characteristics such as some letterpress grades or some starch coated papers, may not cause piling of the type referred to here if the material available for wet picking is soluble enough or "wettable" enough to be carried off by the water or ink with each impression. The cumulative effect from hundreds of sheets is thereby eliminated.

INSTRUMENTATION

The degree of correlation obtained between the results from the two-color press and the "wet thumb tests" warranted designing an "automated thumb." It would also eliminate the need of a picking medium such as ink and reduce the variables to be controlled and maintained. The critical factors to control are water, pressure, time, and release rate. Figure 1 shows the instrument wherein pressure, time, release rate, and moisture are so controlled as to develop reproducible degrees of pick which may be directly related to the piling resistance of the same coated papers on actual offset presses printing two or more colors on successive units.

The present form of this tester consists of a vertically mounted, double-acting air cylinder with an integral four-way solenoid valve having screw-adjustable air-output ports. These ports independently control the rate of descent and return of the piston. The piston is fitted with a metal shoe and the whole piston assembly is supported and aligned on a rigid steel frame over a stainless steel base plate having a

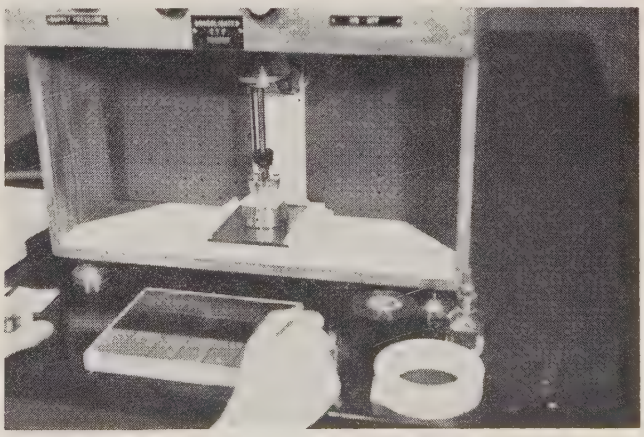

Fig. 4. Dwell time of paper solution and pressure

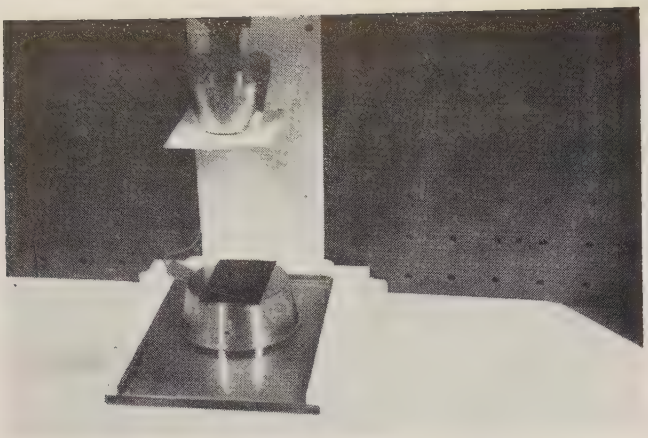

Fig. 5. Pressure release and return

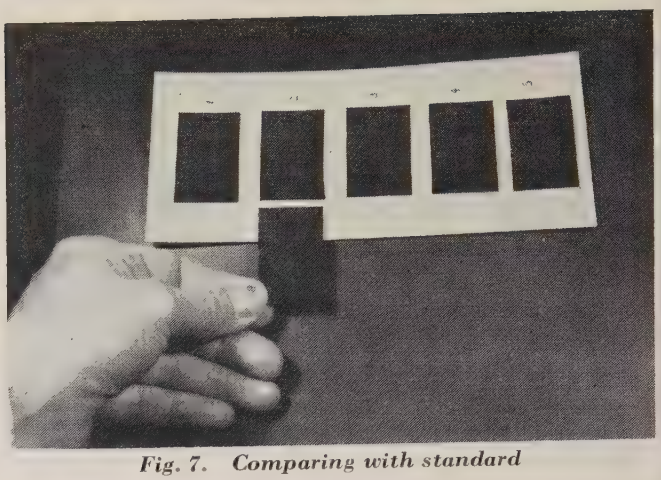

Fig. 7. Comparing with standard

flat machined upper surface. The base plate is firmly affixed to a permanent mounting for accessibility.

Related equipment includes an automatic reset timer having an adjustable 0- to 60-sec. range, a microswitch, an air regulator and gage, and required switches. Also included for penetration testing as a separate function are an electric stop watch or interval timer, current sensitive relay, battery power supply, and microammeter.

The whole instrument is enclosed in a desk-top cabinet mounted on adjustable leveling feet, and requires only a source of compressed air and a single 110 v., 60-cycle outlet for operation.

Figure 2 shows the instrument in operation. Two pieces of black, plastic electrical tape (3M #22) are fitted to the upper metal plate and covered with two-sided pressure sensitive tape (3M #400). The black tape provides some cushion and the pressure sensitive tape holds the sample to be tested. Another piece of the black tape is placed, smooth side up, on the metal base plate and is the surface upon which the pick deposit, if present, will be made.

Figure 3 shows the fountain solution being applied to the tape on the base plate. With the air regulator set at the desired pressure, the air-output ports adjusted for proper down- and return-stroke movements, and the electric timer set for the desired contact interval, a test is started at a push of the start button. The start button energizes the solenoid valve to admit air for the downstroke of the piston. In Fig. 4, contact is made between the paper attached to the stop and the base plate, the drop of moisture is spread by pressure through the contact area, and excess moisture is squeezed out from beneath this area.

At the point of contact, the microswitch starts the electric timer which is so wired that at the end of the predetermined interval it de-energizes the downstroke function of the solenoid valve and energizes the return function. This causes the piston to snap upward with the inrush of air pressure. The timer then re-sets itself of the next test (Fig. 5).

The snap return of the piston with its stop and sample attached is resisted by the forces of adhesion between the moistened paper surface and the surface of the tape on the base plate in such a manner that picks or deposits of coating on this tape may result as seen in Fig. 6. The tape may be visually or optically compared to standards for a numerical or similar grading based on the premises that the greater the degree of relatively water insoluble coating material deposited on the tape by a given set of wet pick test conditions, the greater the piling propensity of the paper in question. The tape itself may then be attached to any report or record desired, since with reasonable care, deposits of coating on the tape will adhere quite readily to form a permanent visual record (Fig. 7).

A test can be completed in less than a minute.

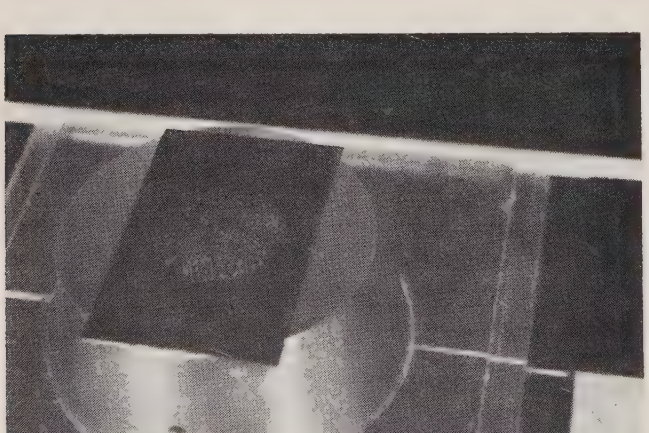

Fig. 6. Coating picks on tape

SUMMARY

Piling as it occurs on the later blankets of a multicolor offset press has been described as a measure of the amount of coating material picked from a paper's surface in such a manner as to upset the existing pigment vehicle balance of an ink.

It was found that by automating the "wet thumb pick test" an instrument could be designed to give a reasonably accurate prediction of the resistance a given sheet of paper would have to the forces causing wet pick during printing.

The instrument features controlled standards of moisture, pressure, and time plus a quick release rate causing a picking force to be exerted on the sample being tested. It is simple to operate, and has potential uses as a penetration and dry pick tester.

Results with the instrument depends upon subjective evaluation, but when used as a comparative control test has correlated well with field trials. Current investigation is being carried on to determine the use of reflectance photometers to replace the subjective ratings.

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Low-Temperature Radiant Heat Drying

HORACE L. SMITH, JR.

Substantial increase in drying capacity of paper machines can be obtained by the application of low temperature, liquid heated radiators. Many materials, including paper, absorb radiant heat almost perfectly when heat is radiated in the long wavelength band (4 to 8 μ) but absorb very little radiant energy in the short wavelength band. The Smitherm process consists of thin, lightweight, sheet metal radiators having a highly emissive surface. The radiators are curved and mounted in a fixed position on the paper machine, with the radiating surface spaced from 2 to 6 in. from the sheet. A high boiling point, nonvolatile, nonflammable, noncorrosive liquid heat-transfer material is heated and circulated through the radiators at practically atmospheric pressure. Approximately $1^1/_2$ lb. of water can be evaporated per square foot of radiator surface per hour. The cost per pound of water removed is substantially lower than by conventional steam driers. Installation of the equipment can be made with practically no shutdown time. The liquid heater can be located several thousand feet from the machine with neglectable loss in transit. Fuel can be coal, oil, or gas.

In the manufacture of paper there is no step in the overall process that is more important or more difficult than the one of removing water from the sheet. As machine speeds increase, drying problems increase at an even faster rate. Although refinements will continue to increase the drying capacity of conventional steam-heated driers, the practical limits in drying rates are fast approaching a point that leaves little opportunity for major rate increases. Steam temperatures are limited by practical pressure considerations. Condensate removal and drier drainage systems have been intensely studied and developed. In fact, every facet of the present basic method of drying has been carefully examined and re-examined so that any major breakthrough in increasing the drying rate of a paper machine must come from a different direction and a new basic approach. The Smitherm process is a basically different means of increasing the drying capacity of a paper machine without the requirement of adding additional driers. The cost per pound of water evaporated will prove to be much less than the cost in the present conventional steam-heated driers.

The Smitherm process will probably find its most immediate application as a supplementary drying means to be used in connection with present paper machines and it does not involve the necessity of making any changes in the present steam-heated driers. It is a supplementary method of adding drying capacity by means of fixed radiators radiating heat to the opposite side of the sheet while it is in contact with the drier surface and which can also be used to dry both the sheet and felts in the draw between adjacent driers and in straight runs of both the sheet and the felts.

RADIANT HEAT

Everyone is familiar with the transfer of heat by conduction and also by convection, but heat transfer by radiation is still a subject that is not well understood by many people and as the Smitherm process, in its application to paper drying, is based on transfer of heat by radiation, a brief discussion of some of the basic principles involved may be helpful in understanding the subject. Radiant heat is energy transferred by electromagnetic vibrations which travel through space at the speed of light, or 186,000 miles per sec. Radiant energy may be regarded as a form of wave motion with the length of the waves varying according to the

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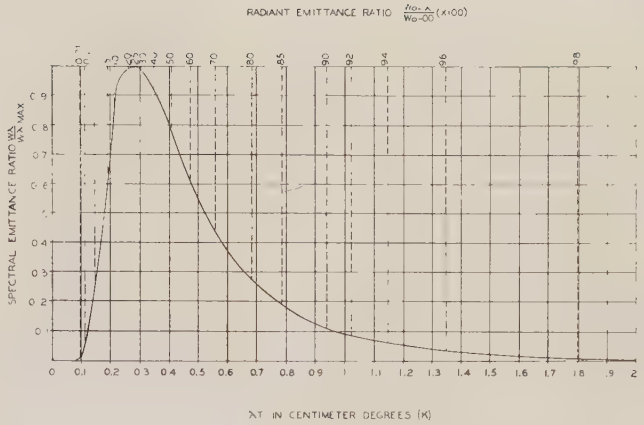
RADIANT EMITTANCE RATIO $\frac{W_{\lambda T}-A}{W_{\lambda T}-00}(\times 100)$
SPECTRAL EMITTANCE RATIO $\frac{W_\lambda}{W_\lambda \text{ MAX}}$
λT IN CENTIMETER DEGREES (K)

Fig. 1

temperature of the radiating source. Max Planck formulated the quantum theory and Fig. 1 is a graphical plot of the energy distribution, a ratio plot showing the spectral emittance ratio of energy (*1*). The pracitcal significance of Planck's equations is that for any given black body temperature, most of the energy is emitted in a rather narrow wavelength band. Some energy, however, is given off in bands of both shorter and longer wavelengths and, at the point of maximum energy radiation, approximately 25% falls below a given wavelength with 75% of the energy at longer wavelengths. The higher the temperature of the radiator the shorter the length of the waves given off.

For any given temperature, a hot solid body sends out radiations over a wide range of wavelengths but the intensity of the radiation differs greatly for the various wavelengths and the largest amount of energy is radiated at a wavelength band which corresponds to a given temperature.

As radiant energy is transferred by wave motion, at the speed of light, and does so through a vacuum as well as through some gases and liquids, the distance that radiant energy travels does not change the amount or intensity of the radiant heat, unless it is passing through some absorbing medium.

Wien's displacement law specifies the position of peak energy relative to the wavelength and states that the maximum value for radiation intensity moves with increasing absolute temperature to the shorter wavelengths. In other words, with an increase in temperature the peak of the distribution curve moves to shorter wavelengths.

QUANTITY OF HEAT RADIATED

The Stefan Boltzmann law defines the total amount of energy radiated from a body at a given temperature and states that the amount of energy radiated varies as the fourth power of the absolute temperature. In the application of the Stefan Boltzmann law, it is necessary to know the surface temperature and the condition of the surface, or the emissivity coefficient. In the equations of Max Planck there is involved a concept of the "black body," which is defined as a body which absorbs all radiation of all wavelengths falling upon it and reflects none. Such a body also radiates the largest amount of energy possible at any given temperature and the radiation from a black body is called "black body" radiation, even though it contains much light. The emissive power of a black body depends on its temperature only. By the same token, a black body emits the maximum amount of radiant energy and the emissivity coefficient is unity. It is not possible to have a radiating surface that has an emissivity coefficient of 1.00, but it is possible to obtain emissivity coefficients in the order of 0.95. A surface having an emissivity of 0.95, however, would radiate 95% of the energy that a theoretically perfect black body would give off. A highly polished reflecting surface, such as a mirror or polished metal surface, reflects most of the radiant energy falling upon it. Therefore, its emissivity is very low and can be in the order

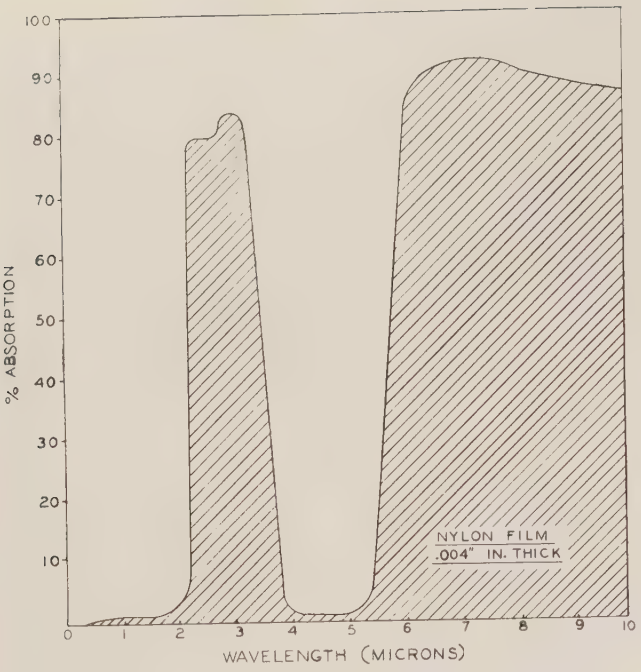

Fig. 2

of 0.05, which means that it would radiate only 5% as much energy as a black body at the same temperature.

An examination of the Stefan Boltzman law shows that an increase in the temperature of the radiating surface causes energy to be given off at a rapidly increasing rate as a fourth power function of the absolute temperature. Yet when we attempt to apply this in practice, it becomes apparent that in order to get a satisfactory quantity of heat radiated per square foot of surface it is necessary to obtain temperatures much higher than can be provided by steam at more or less conventionally acceptable pressures. Steam has always been considered the most feasible source of heat for industrial needs but it does not provide a good source of energy for radiant heating. If an attempt is made to get higher temperatures by use of steam, an inspection of the steam table vividly points up the fact that with an increase in pressure, the temperature increases at a diminishing rate. In order to obtain a temperature of 500°F. it is necessary to have a pressure of almost 700 lb. and in order to get a surface temperature of 600°F., it is necessary to more than double the pressure, as a pressure of approximately 1600 lb. is required. A surface temperature of 600°F. and an emissivity coefficient of 0.95 radiates more than 2000 B.t.u. per sq. ft. per hr., whereas steam at 100 lb. gage pressure has a temperature of 338° and radiates only about 650 B.t.u. per sq. ft.

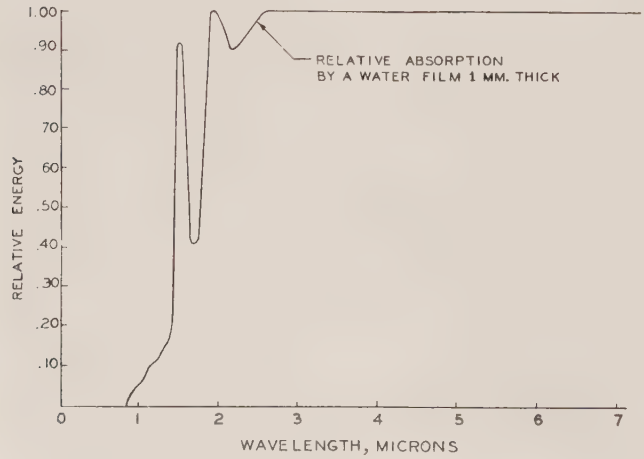

Fig. 3

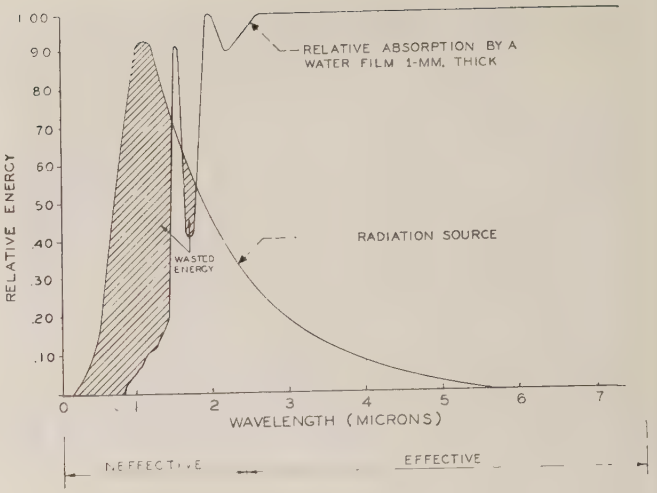

Fig. 4

per hr. It is, therefore, necessary to have surface temperature much higher than can be provided by conventional steam sources if radiant heat is to be considered practical.

ABSORPTION OF RADIANT HEAT

Probably the most obscure and least understood phenomenon in connection with the application of radiant heat is the way that radiant heat is absorbed or rejected from the host material that is to be heated. A typical example will explain this point. Figure 2 shows the rate of absorption of radiant energy by a film of nylon 0.004 in. thick (*2*). The shaded area shows the per cent of absorption and it is interesting to note that energy radiated at a wavelength of 6 μ or longer is absorbed quite readily by the nylon. Yet if the peak of the radiant energy was at a wavelength of $4^1/_2$ μ, very little of the energy would be absorbed. If it were possible to radiate energy in a very narrow band of from 2.5 to 3.5 μ then, again, the rate of absorption would be satisfactory.

Again referring to the curve showing distribution of radiant energy in Fig. 1, it is apparent that it is not possible to obtain a radiant energy distribution curve that would fall within the steep and narrow absorption area. Another example is shown in Fig. 3, which illustrates the radiant energy absorption curve of a film of water 1 mm. thick. It is to be noted that energy having a wavelength less than 2 μ is very poorly absorbed.

Figure 4 shows the same absorption curve for a film of water. Superimposed over this curve is the energy distribution curve with its peak at 1 μ. The shaded area is a rough indication of the amount of energy available but not usable because it is not absorbed by the film of water.

Figure 5 again shows the absorption curve of a film of water

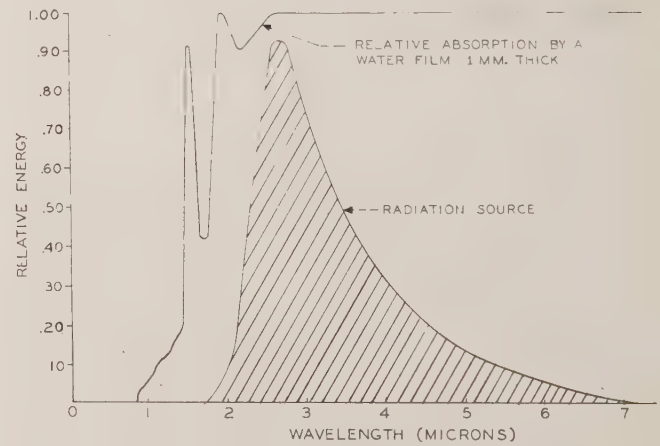

Fig. 5

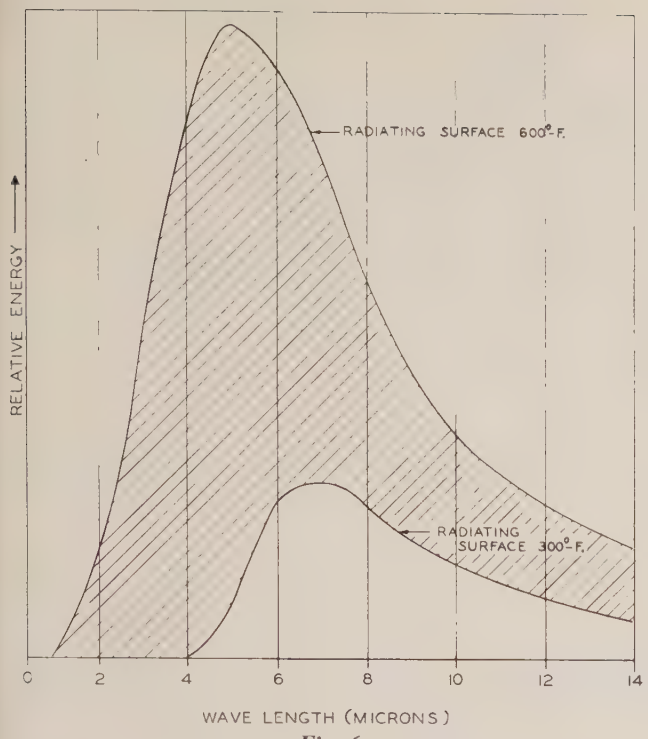

Fig. 6

and the energy distribution curve with a peak value of 2.5 μ wavelength. It is immediately apparent that all of the energy is now being absorbed by the film of water.

Figure 6 shows the relative amount of energy radiated from a surface at 300° and one at 600°. It also shows the shift to the shorter wavelength of the peak of the energy curve with an increase in temperature. At 300°F. the maximum is 6.9 μ and at 600° the maximum is 4.93 μ.

Every material has an absorption curve of somewhat different configuration, as shown in Figs. 2 and 3, but all materials have one common characteristic. In the very short wavelength band, the absorption of radiant energy is very poor.

WAVELENGTH OF ELECTRO-MAGNETIC SPECTRUM

Figure 7 shows a logarithmic scale covering the wavelength of the entire electromagnetic spectrum, from the cosmic rays in the extreme short wavelength band to the long electrical oscillations. The shaded area on this scale between the ultraviolet and the infrared rays has been expanded to show a portion of the scale from 0.1 to 20 μ and it will be noted that the entire visible spectrum is included in the narrow band of 0.4 to 0.7 μ. This in turn has been expanded to show the color spectrum from the ultraviolet to the infrared.

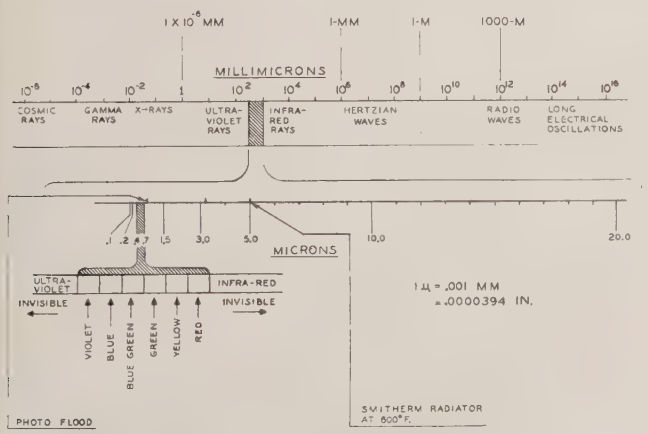

Fig. 7

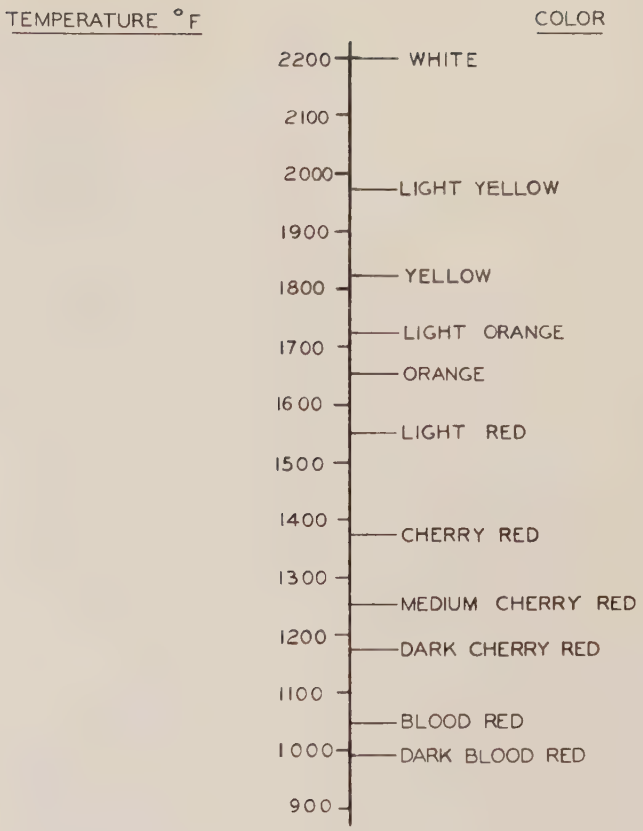

Fig. 8. Relation of color to temperature of iron or steel

The relation of color to temperature is shown in Fig. 8.

In order to realize the tremendous range of wavelengths in the electromagnetic spectrum, it is to be noted that radio waves are in the range of hundreds of meters, but light waves and infrared waves are so small that they are measured in a much smaller unit, the Angstrom, which is only 1 ten billionth of a meter. One micron is 1 millionth of a meter. Infrared rays cover a range of from 0.8 to 2.7 μ (8000 to 27,000 A.) and the near infrared covers a range of 2.7 to 600 μ (27,000 to 6,000,000 A.).

Figure 9 is a nomograph for determining the rate of heat dissipation from a hot surface in a temperature range from 0 to 800°F. and for different emissivity coefficients. The example illustrated shows the amount of energy radiated from a surface at 600°F. with an emissivity coefficient of 0.95. On the temperature scale is also noted the wavelength that corresponds to the peak of the energy distribution curve.

There are many installations of electric radiant heaters and these are of various forms, from rodlike elements that operate at a dull red temperature on up through the higher temperatures of incandescent lamps fitted with metal foil reflectors and quartz lamps that operate at extremely high temperature. The information shown in Figs. 7 and 8 clearly shows that all of these radiators are operating at temperatures that are giving off the greatest amount of energy in the very short wavelength band and, therefore, will not be efficiently absorbed by many materials.

When heat is given off from a radiant source in the longer wavelength bands, it is readily absorbed by practically all material and color becomes unimportant. In the very short wavelength band, black or dark colors absorb radiant energy at a much greater rate than white or light colors but in the long wavelengths practically all materials are color blind and all have a very high rate of absorption. White paper has an absorption coefficient of only 0.25 at 0.95 μ.

NET AMOUNT OF HEAT TRANSFERRED

Figure 10 illustrates another important fact in the application of radiant heat. Every object or body is giving off

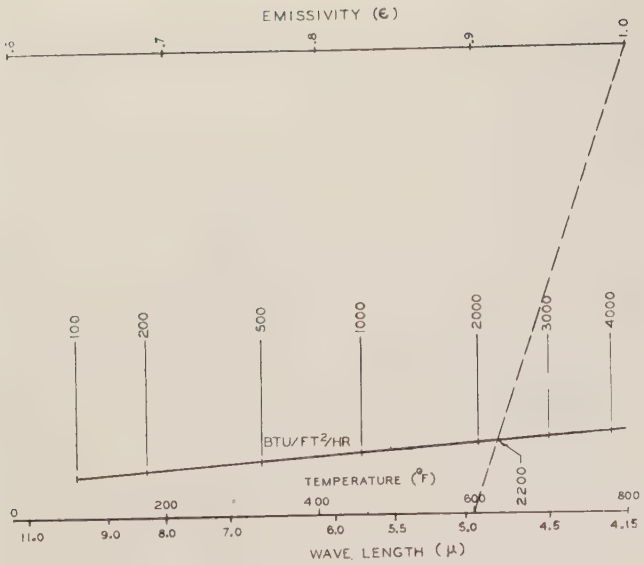

Fig. 9. Nomograph-radiant heat transfer

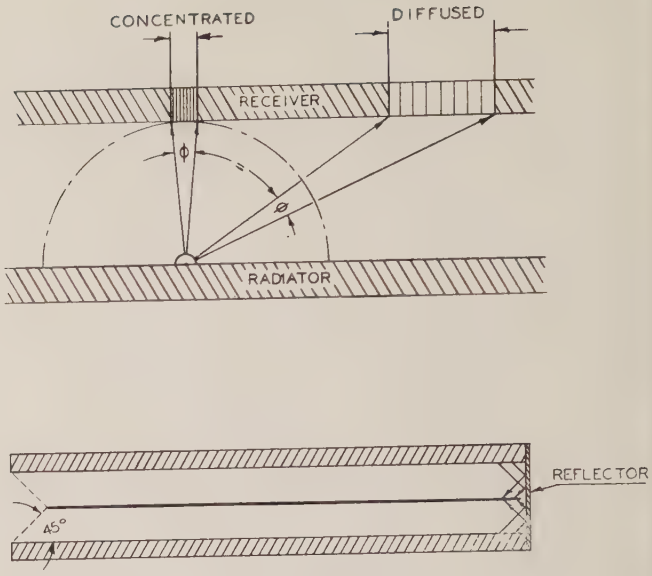

Fig. 11

radiant heat in proportion to its absolute temperature and the surface emissivity coefficient, and at the same time it is absorbing radiant heat from all surrounding bodies. The net rate of change is the difference between the amount of energy given off and the amount of energy received. Figure 10 illustrates this point by assuming radiator R1 at a surface temperature T1 of 600°F. and an emissivity coefficient of 1.00. This surface gives off energy at the rate of 2150 B.t.u. per sq. ft. per hr. Radiator R2 has a surface temperature T2 of 210°F. and an emissivity coefficient of 1.00 and therefore gives off heat at a rate of 350 B.t.u. per sq. ft. per hr. The net transfer of energy is the difference between these two rates, or 1800 B.t.u. per sq. ft. per hr.

In Fig. 11 an attempt is made to show that radiant energy is dissipated equally in all directions. The top drawing shows a radiating surface and for every point on this surface, energy is being radiated equally in all directions. If the point was at the center of a hemisphere, every unit area on the interior surface of the hemisphere would receive the same amount of radiant energy. If energy is being radiated to a surface parallel to the radiator, it is apparent that more energy is concentrated at a point at right angles to the radiating surface than is being received at a more distant point. This effect is important when heating a sheet or web or any surface parallel to a radiator because it shows that if the surface being heated is the same width as the radiator and that the edge of the sheet would receive less energy than areas toward the center of the radiator, because some of the energy from the radiator would be dissipated beyond the sheet being heated. The lower drawing shows that as the distance between the radiator and the sheet being heated is increased, the width of the radiator should be increased in proportion, and should never be less than required to provide an angle of 45° or less between the edge of the radiator and the edge of the product. The right side of this same drawing shows an alternate method that can be used to reflect the radiant energy back to the product, thereby eliminating the need of making the radiating surfaces wider than the product.

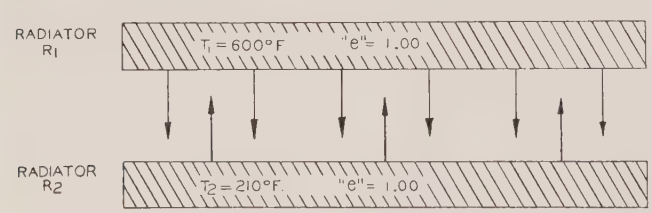

Fig. 10

Energy radiated from R1— 2150 B.t.u./sq. ft./hr.
Energy re-radiated from R2— 350 B.t.u./sq. ft./hr.
Net energy transfered to R2— 1800 B.t.u./sq. ft./hr.

COMPROMISE OF RADIATION VERSUS ABSORPTION

From the facts brought out in the preceding discussion, it becomes clear that heating from a radiant source can be very effective if the subject is properly understood and carefully analyzed. The Stefan Boltzmann law indicates that high temperatures should be used because the amount of energy given off per unit area increases rapidly, as the rate is a function of the fourth power of the absolute temperature. An examination of the rate of absorption of radiant energy by most materials shows that energy radiated in the short waveband is not readily absorbed by the material, therefore, a compromise is necessary in order to obtain the highest practical over-all efficiency. Dimensional analysis of the problem indicates a temperature range between 500 and 600°F. as providing a satisfactory rate of heat output and at the same time keeping the greatest amount of energy in a wave band that is absorbed by the host material at a satisfactory rate. In order to obtain temperatures within this range, it is necessary to find a practical and economical source of heat other than steam. It is also necessary to find a simple, practical, and economical form of radiator having an emissivity coefficient of the highest possible value.

The Smitherm process is not a machine or a single piece of apparatus but is a system engineered to satisfy the optimum conditions indicated by the above facts. A high boiling, nonflammable, liquid heat transfer media is used. This material is noncorrosive, has low viscosity at operating temperatures, and possesses good heat transfer characteristics. It can be heated in an open container at atmospheric pressure to 600°F. The liquid is heated in the equivalent of a water tube boiler, although boiling does not occur. A centrifugal pump circulates the liquid through the heater then through the radiators and back through the heater. An open or vented expansion tank is used, therefore the only pressure in the system is the hydrostatic pressure of the liquid due to the head of the elevated expansion tank plus pipe friction in the system. The overall arrangement is identical to a forced circulation domestic hot-water heating system using an overhead vented expansion tank.

RADIATORS

A special type of radiator has been designed that provides light weight and low cost, can be made in flat panels or curved to any required configuration, and is obtainable in

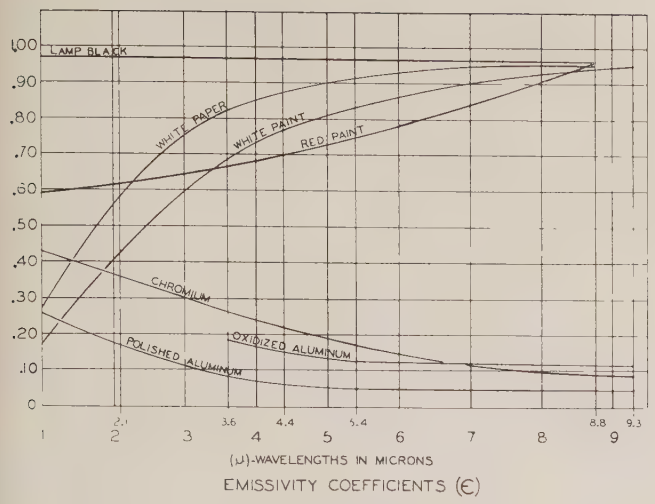

Fig. 12

practically any size. In addition to a suitable radiator, a surface treatment has been developed that provides an emissivity coefficient of 0.95. The emissivity factor E is a very important consideration in the practical application of radiant heat. Surface conditions after the emissivity coefficient and changes in temperature also cause a change in the emissivity factor. A white pigment might have an emissivity coefficient of 0.08 at 5000°F. and a value of 0.71 at 750°F. and at 125°F. the coefficient would be increased to 0.89. It is this great change in the emissivity and absorption characteristics of a surface that provides such a high rate of energy absorption at the lower temperatures (and longer wavelength) for all surfaces, regardless of color, and also explains the wide variation in absorption characteristics of surfaces of varying colors at the higher temperatures.

ABSORPTION CHANGES WITH WAVELENGTH

Figure 12 shows how the emissivity of several materials change with wavelength. It is to be noted that emissivity and absorption are the same. The emissivity curve for lampblack is almost constant and uniformly high; whereas, white paper has a very low absorption coefficient at 1 μ, but from 3.6 μ and higher the absorption is excellent. In fact at 5 μ (approximately 600°F.) the absorption of white paper is almost as good as lampblack. Another interesting point is the relative shapes of the absorption curves of different materials. Red paint has a relatively flat curve compared to white paint and paper. Chromium and aluminum show a decline in emissivity with increase in wavelength and even an oxidized aluminum surface makes a good reflector in the longer wavelengths.

A fact little understood is that although snow reflects light to a high degree it absorbs radiant energy in the long wavelengths almost as perfectly as a black body.

In Fig. 13 the upper drawing shows in very elementary fashion the essentials of a Smitherm plant. The liquid heater can be fired with the most economical available fuel and can be located practically any distance from the paper machine. Loss of heat in transmission is very low, less than 1% per thousand feet of pipe. Due to the low viscosity of the heating liquid, pumping losses are very low and liquid velocities can be quite high, thereby permitting the use of small size pipe. As the liquid is noncorrosive and operates under practically no pressure, standard schedule 40 steel pipe will last indefinitely.

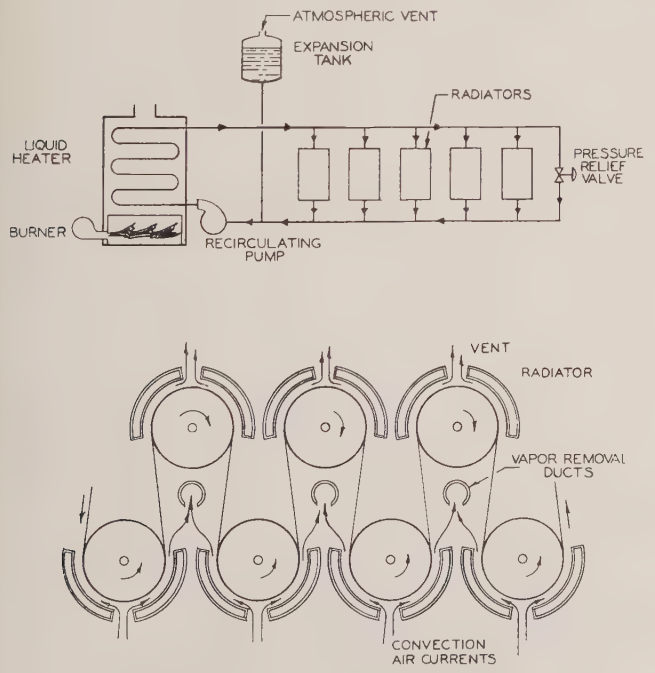

Fig. 13

Again referring to Fig. 13, the lower part of the drawing shows diagrammatically how the radiators would be fitted around the driers of a paper machine. Insofar as the regular driers are concerned, no change would be made. The Smitherm radiators would be fitted so as to obtain the maximum amount of radiating surface, both around the driers and on the draw or straight runs. The arrangement shown in Fig. 13 is not to scale because the radiators themselves are actually only 1 in. thick.

Figure 14 shows the general construction of both the single and double embossed radiators. The single embossed construction is used when it is only necessary to radiate from one side whereas the double embossed radiator is used when radiation from both sides of the radiator is required.

In Fig. 15 the general arrangement is shown whereby a sheet or web is heated by single embossed radiators on the outside and a double embossed radiator between the two runs of the sheet. The cross-hatched portion on the outside faces of the single embossed radiators in Fig. 15 represents insulation.

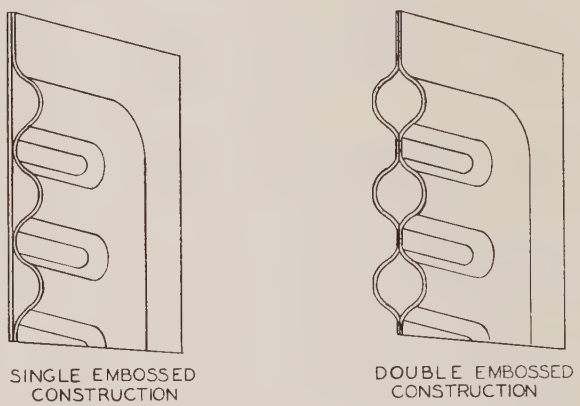

Fig. 14

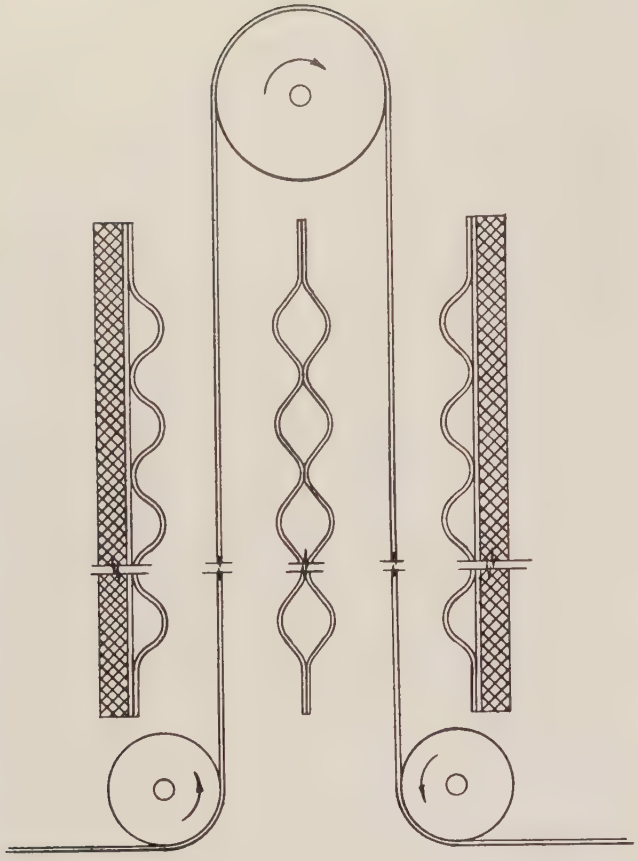

Fig. 15

TEMPERATURE CONTROL

Although the temperature of the heating liquid can be accurately controlled, an additional refinement in the way of precision temperature control can be obtained by an arrangement shown in Fig. 16. This shows a radiator heating a sheet. Between the radiator and the product are light sheet metal vanes with highly reflective surfaces, such as polished aluminum. These vanes are arranged similar to venetian blind slats so that one operating rod can rotate a bank of vanes. To the left of the drawing, the vanes are shown in a position to permit free flow of radiant energy. The center section shows the vanes moved through a small angle whereby they intercept a small amount of the radiant heat and to the right the vanes are shown shingled or overlapped, thereby reflecting all of the heat to the original source and cutting it off from the product.

In Fig. 17 is shown an arrangement whereby separately controlled temperatures can be maintained in radiators or other devices independent of the temperature of the main supply. In the example used in the drawing, the temperature sensing element on the radiator acts through the controller to continuously modulate or adjust the position of the three-way diverting valve so that a part of the liquid circulated through the radiator is returned to the suction side of the pump and only a sufficient quantity of high temperature liquid is taken from the supply to maintain the required temperature in the radiator. This principle of control can be used on calender stacks, radiators, or any other devices

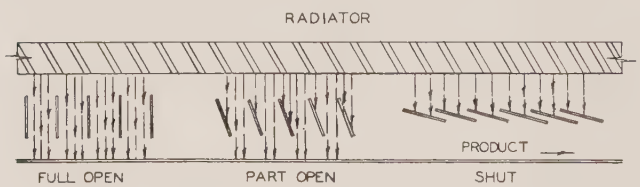

Fig. 16

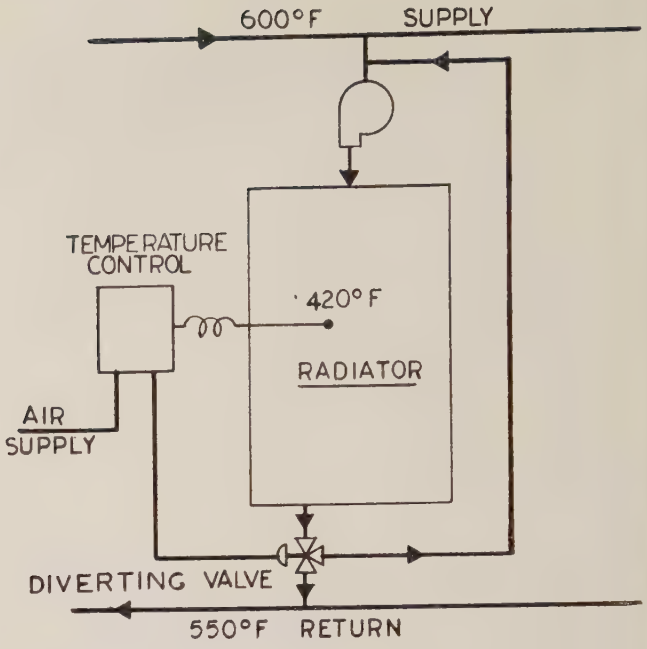

Fig. 17

that are to be operated at a temperature that is lower than the temperature of the supply source.

COMPARISON OF OTHER METHODS

In considering the practical application of the Smitherm process to a paper machine, it is necessary to examine the methods and limitations of present practice. In the manufacture of paper, the basic method of drying the sheet is of passing the wet sheet around steam heated drums. There are many variations in details, such as the several systems for removing the condensate from the drier drums, various methods of increasing air velocity over the face of the sheet, vapor removal devices, etc. One method that is radically different from the conventional system is the Minton vacuum drier, which employs a vacuum tight enclosure for the entire drier with the pressure within the enclosure reduced in order to lower the boiling point of the water in the sheet. No air is circulated through the machine, as the entire space within the vacuum tight enclosure is filled with water vapor. This must be removed at the same rate at which it is generated, otherwise it would not be possible to maintain the required vacuum.

In both the conventional drier and the Minton vacuum drier, all the heat required for the evaporation of water is transferred by conduction and the rate of transfer is limited by the relatively small difference in temperature between the surface of the drier drum and the temperature of the sheet. Physical limitations make it difficult to obtain higher drum surface temperatures. As the rotational speed of driers increases, centrifugal force causes the condensate in the drier to take the form of a hollow cylinder of water. To date, no drier drainage system has been completely successful in eliminating this cylinder of condensate. The steam condenses on the interior surface of the water and the film coefficient at this point is quite high, but heat has to be conducted through the wall of water in order to reach the interior face of the drier shell. The conductivity of water is 0.395 B.t.u. per hr. per sq. ft. per degree per ft., whereas the conductivity of cast iron or steel is 26. Therefore, the conductivity through the water is at a rate of only $^1/_{65}$ of the rate through either cast iron or steel. Heat transfer by convection has little effect at this point because, due to centrifugal force, the highest temperature of the water is at the steam water interface and the lowest temperature of the water is at the interface of the condensate and the drier shell, due to the fact that the cooler water is heavier and centrifugal

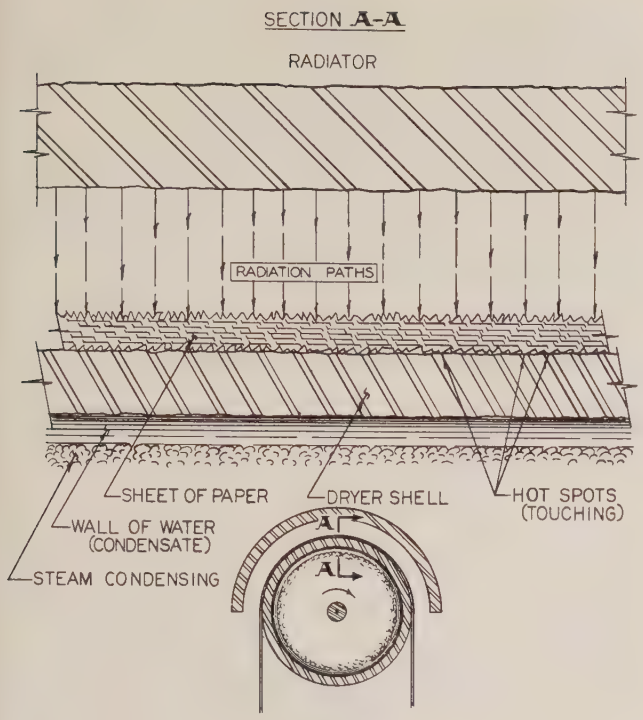

Fig. 18

force maintains a natural thermal gradient throughout the thickness of the wall of water.

In order to obtain higher surface temperature at the contact surface between the drier and the sheet, it is necessary to increase steam pressure and, as pressure increases, it is necessary to increase the thickness of the drier shell in order to provide the required increase in strength. As a thermal gradient must exist throughout the thickness of the shell, the net result is that a substantial increase in steam pressure results in a very small increase in drier surface temperature.

The only two ways in which heat can be transferred from the surface of the drier to the sheet are by radiation and conduction. The amount of heat transferred by radiation is of little importance, being approximately 200 B.t.u. per sq. ft. per hour. This value is based on an assumed drier surface temperature of 220° and an emissivity coefficient of 0.5. This would represent the maximum theoretical rate, assuming that the sheet was at a relatively low temperature. If the temperature of the sheet is 180 to 200°F., there is practically no heat transfer by radiation because the Stefan-Boltzmann law shows that the net transfer of energy by radiation between two surfaces is equal to the amount of heat given off by the highest temperature surface less the amount of heat given off by the lower temperature surface. The emissivity coefficient of a smooth semipolished drier drum would probably be 0.55 and the emissivity coefficients of most paper products are 0.7 to 0.92.

Transfer of heat by conduction between the surface of the drier and the sheet is a complex process involving the presence or absence of an air film separating the sheet from the drier surface; pressure normal to the surface of the sheet, when thermal conductivity of the sheet at the instant is considered, and the actual percentage of contact area. A sheet of paper is not a smooth surface but consists of a series of high and low points, such as ridges and valleys or peaks and depressions. Therefore, a relatively small percentage of the total area of the sheet is in intimate heat transfer contact with the drier. This is, of course, why crepe papers are so difficult to dry. One of the important functions of drier felts is the relatively high tension maintained in the felt which increases the total pressure normal to the surface of the sheet and forces a larger percentage of the sheet in intimate heat transfer relationship with the drier. As the diameter of a drier is increased, the unit pressure normal to the surface of the sheet is decreased, assuming the same tension exists in the sheet and/or the felt.

Figure 18 includes the small drawing showing a conventional steam heated drier with a curved radiator fitted around the drier and spaced away from the sheet. The large scale drawing shows a section through the radiator, sheet, and drier shell. Steam is condensed on the inner surface of the cylinder of the condensate in the drier and heat flows by conduction through the condensate, through the drier shell, and then into the sheet of paper. The sheet of paper is shown grossly exaggerated in thickness in order to emphasize the surface roughness. Photomicrographs of any sheet of paper will point up the fact that the actual area of high pressure contact between the surface of the drier and the sheet is relatively a small percentage of the total area.

In conventional drying, moisture loss occurs mainly by diffusion, because at temperatures below the boiling point evaporation can take place only in the presence of air. If a saturated film of air is in contact with the sheet, there will be no additional loss of moisture from the sheet until this saturated film is removed and an unsaturated film takes its place. For this reason, it is necessary to maintain a relatively high velocity of air over the surface of the sheet in order to continuously remove the saturated film and replace it with a drier film. Unless this air is heated to approximately the same temperature as the sheet, the colder air will have a chilling effect on the sheet, thereby reducing the rate of diffusion.

Apparently, as a sheet of paper comes in contact with the drier surface the relatively cooler sheet must first be reheated to the point of vaporization and, due to the higher vapor pressure existing at the contact surface, moisture tends to migrate to the outside surface. However, if the contact time on any one drier is insufficient for an appreciable amount of moisture to leave the outer surface, part of this moisture can be recondensed, thereby forming a higher moisture concentration on the outside portion of the paper. The next successive drier that contacts the sheet will be on the opposite side of the sheet. Therefore, part of the moisture in the sheet can take a zigzag path, moving first from near one surface, then to the opposite surface, etc. Apparently, the greatly increased unit rates on Yankee driers are caused by the much longer and more intimate contact time between the paper and one drying surface, which permits the dissipation of the air film trapped between the sheet and the drier surface and also eliminates the zigzag pattern of moisture migration within the sheet itself.

By using Smitherm radiating panels fitted around the driers and also over and under straight runs of the sheet, a substantial increase in drying rates can be obtained. Each square foot of radiating surface provides over 1500 B.t.u. per sq. ft. per hr. of additional heat into the sheet and drier cylinder, which not only provides for the evaporation of at least $1^1/_2$ lb. of water per square foot per hour in addition to the water evaporated by the driers but actually increases substantially the drying rate of the driers themselves. This increase in drying rate is due to the fact that without the Smitherm radiators the air that is circulated in and around and over the sheet is at a lower temperature than the boiling point to chill the sheet. With the Smitherm radiators, there is no necessity for circulating air and the radiators not only prevent loss of temperature on the outside of the sheet but raise the temperature of the sheet to the boiling point.

When heat is transferred by conduction and passes through an interface between two dissimilar materials, this interface usually forms a heat barrier. In the case of the interface between a sheet of paper and the surface of a drier, an insulating film of air is carried along with the sheet and the faster the speed of the sheet, the greater the amount of air caught in the nip between the sheet and the tangent point of the drier. Obviously, this film of air will be gradually dissipated by being forced through the sheet, but due to the short time

contact between the sheet and the drier, the air is never entirely eliminated.

Higher rates of heat transfer between the drier surface and the sheet could be obtained if it were possible to increase the pressure normal to the surface of the sheet. It is believed that this is one of the most important functions of drier felts because of the great increase in tension maintained in the felts as compared to the maximum tension possible in the sheet. The pressure normal to the surface of the sheet is directly proportioned to the tension in the sheet and in inverse proportion to the radius of the drier cylinder. Therefore, as the diameter of the drier is increased, the force or pressure normal to the surface decreases.

Heat transferred by radiation to the sheet does not have the large temperature difference between the hills and valleys,

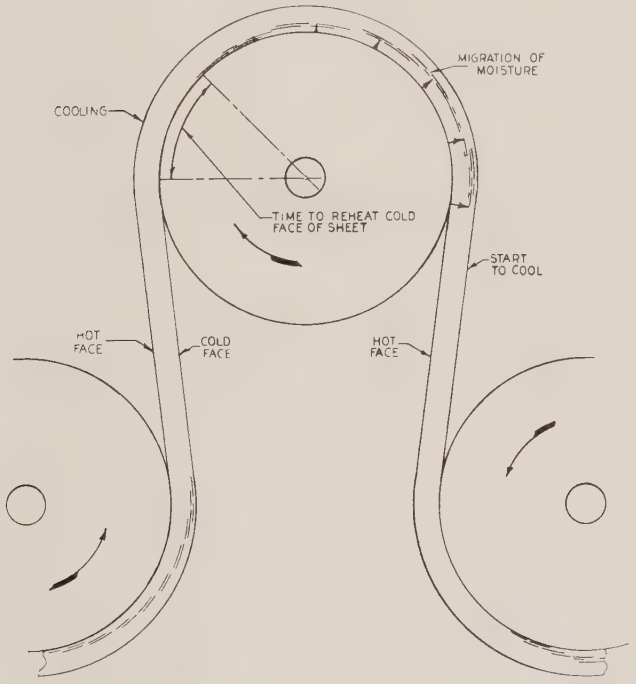

Fig. 19

because radiant energy penetrates into the pores of the surface and the areas in the valleys or depressions receive heat at the same rate as the ridges or peaks. The most spectacular demonstration of this effect is the decrease in drying rate on crepe papers with conventional driers and the actual increase in drying rate with radiant heat for the same product due to the increase in surface area available for absorption of radiant energy. Figure 18 shows the point mentioned above.

Figure 19 shows a condition that apparently exists, especially in thick sheets, such as absorbent papers, etc. As the sheet contacts a drier, the inside of the sheet requires a certain amount of contact time to be reheated, due to the cooling effect in the draw. Moisture starts to migrate from the inside face of the sheet to the opposite side and part of the moisture is liberated through the surface. But a considerable amount has not reached the outside surface by the time the sheet leaves contact with the drier, causing the moisture to be condensed due to the cooling effect of the draw. It therefore remains within the structure of the sheet, taking a more or less zigzag path within the sheet as the two sides of the sheet are in alternate contacts with successive driers. If radiant heat was being added to the sheet while it was in contact with the drier, the arc of contact required to reheat the sheet would be greatly reduced and moisture would start to leave the surface much earlier.

The cost per pound of water evaporated by the Smitherm radiant heat process is far less than the cost per pound by steam used in conventional driers. The liquid heater that converts the heat in the fuel into sensible heat in the heat transfer liquid is comparable in efficiency to the best designed steam generators of equal capacity, therefore, the available heat at the prime source is of equal cost per B.t.u. The heat lost in transit in a Smitherm installation is far less than the losses incidental to heating by steam. In conventional driers, the efficiency or pounds of steam per pound of water evaporated naturally vary through wide limits, depending on many factors. Possibly 1.5 to 1.75 lb. of steam per pound of water evaporated would be a fair average. Steam at 30 lb. gage pressure has a total heat of 172 B.t.u. per lb. Considerable loss occurs in the drier drainage system and in returning the condensate to the boiler. It is reasonable to assume that the average total heat in the condensate is 100

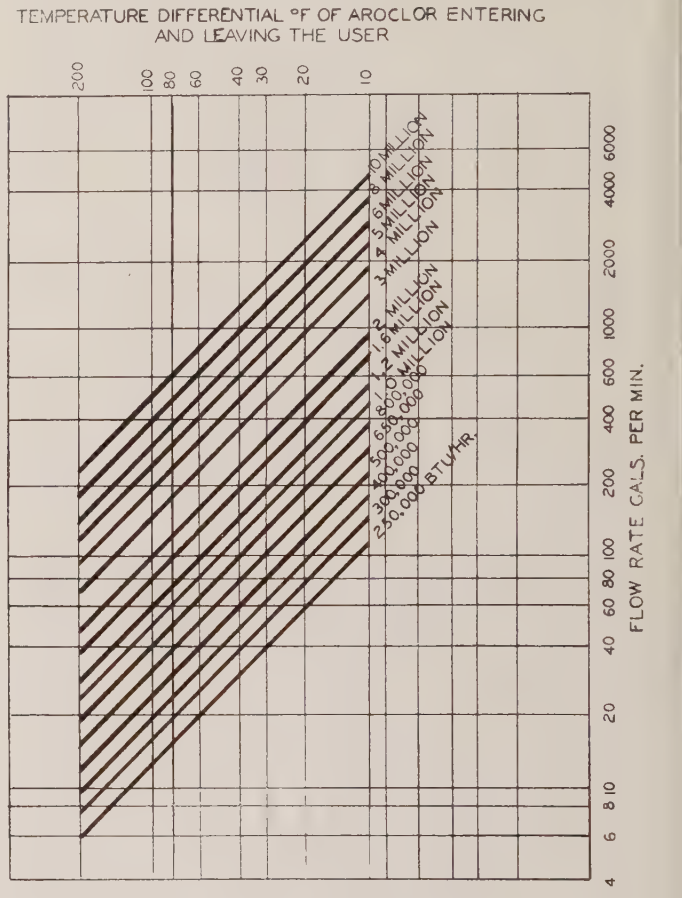

Fig. 20

B.t.u. per lb., or a net loss of 1072 B.t.u. per lb. The latent heat of vaporization at 212° is 970 B.t.u. per lb. Therefore, if an average of 1.62 lb. of steam per pound of water is evaporated, the total heat content of the steam leaving the boiler required to evaporate 1 lb. of water would be 1898.6 B.t.u. and the heat in the condensate returned to the boiler (assuming 100% recovery of condensate) is 162 B.t.u. Therefore, the net heat loss is 1736.6 B.t.u. required to supply 970 B.t.u. to evaporate 1 lb. of water or a ratio of 1.8 to 1.

QUANTITY OF HEATING LIQUID

Figure 20 shows a series of curves plotted on log scale that gives values of liquid capacity plotted against temperature drop and flow rates. Taking an example of a total temperature drop through the system of 50°F. and a heat load of 10,000,000 B.t.u. per hr., the flow rate would be approximately 1000 g.p.m.

The viscosity of the liquid is relatively low, 0.5 cp. at 550°F., and this is approximately the same viscosity as

water at 125°F. This permits high velocities to be maintained in the system without incurring high pumping costs.

Although the design of a Smitherm installation does not deviate from sound standard engineering practice, there are many special details that must be considered. In the selection of the liquid heater, a heater is necessary that will prevent local overheating of the liquid, thus uniform and very high velocities must be maintained in all of the areas within the heater that are exposed to either radiant heat or hot products of combustion. The piping system required for distribution of the heating liquid must be carefully designed in order to prevent excess strains or distortion caused by expansion due to the great range of temperature to which the piping system is subjected. Thermal insulation must be carefully selected on the basis of durability, effectiveness, and economics. Pumps used for circulating the heating liquid must be selected carefully in order to maintain leaktight shaft seals at the operating temperatures and must also have proper performance characteristics to permit cold starting when the viscosity of the heating liquid is much greater than at working temperatures. Rotating joints for passing the heating fluid into and out of rolls such as calenders, heating drums, etc., must be selected with knowledge and experience. Control valve selection must be based on experience and complete understanding of the operating conditions. In other words, the entire design of a Smitherm plant requires both sound engineering and a wide background of experience in the peculiarities of high temperature engineering.

SUMMARY

In summary, the salient points in the Smitherm process are as follows:

Drying capacity of a paper machine can be substantially increased without adding drier sections to the machine and at much less cost than by obtaining the same additional drying capacity by means of adding conventional driers.

By careful planning and scheduling, a Smitherm installation can be made on an existing paper machine with practically no down time of the machine.

The operating cost per pound of water evaporated is much less than the cost of evaporation by conventional steam driers. The only losses are radiation losses in the distribution system and these are almost insignificant. There is no loss of heat in flashing high temperature condensate or in loss of condensate.

No expensive additions have to be made to the paper machine such as moving calender stacks, rewind equipment, additions to drives, building extensions, etc.

Precision temperature controls throughout the entire drying cycle.

All of the operational advantages of a vacuum system, such as the Minton drier except for the lower temperature, are provided without the cost of vacuum enclosure and with the added advantage that the machine is entirely accessible to usual operating procedures.

The Smitherm process is a carefully engineered development based on sound engineering fundamentals in which high efficiency is obtained by a thorough knowledge of the heat absorption characteristics of the product, matched against proper radiating wavelength of the heat source, utilizing conventional low cost fuels.*

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* This system is covered by pending patent applications.

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Some Chemical Aspects of Towel Manufacture

J. P. BELL

The problems encountered over the past five years in the manufacture of paper towels at the Westminster Paper Co. are described from the technician's viewpoint. The main topics deal with the preparation, selection, and application of eight major chemicals and the machine problems they have created. The problems encountered in the various manufacturing steps are outlined, starting with the furnish and ending with the finished paper. It is concluded that careful control of the chemical furnish is essential and that greater emphasis should be placed on studying the colloidal conditions existing in paper machine systems.

This paper describes the technical aspects of towel manufacture at the Westminster Div. of the Westminster Paper Co. The main topics outline the problems encountered in preparing and testing the chemicals which make paper strong while wet and the machine room problems created by these additives.

The eight major chemicals which have been used in the past five years are: a melamine resin, two cationic urea-formaldehyde resins (hereafter referred to as resins A and B), two wetting agents, hydrochloric acid, sulfuric acid, and alum. The melamine was discarded in 1954 due to its high cost compared to the other compounds.

RESIN PREPARATION

The melamine pumping system was used for the other resins until 1956. Before considering the limitations of this handling system, note that a mixture of melamine and urea resins will form a tacky precipitate, therefore the equipment must be thoroughly cleaned.

This pumping equipment was built to deliver solutions of 6% solids, so resins A and B had to be diluted with water from 34.7 and 32.5% solids, respectively, to approximately 8.7% solids.*

This dilution was not desirable because the storage life was too short and the resin sometimes precipitated during makeup. Gellation during dilution was caused by the water temperature falling to 5°C. or lower when the critical solution temperature of an 8% solids solution was 9°. In fact, the critical temperature was higher at this dilution than at either 3 or 12%. The storage life was short because the gellation temperature increased more rapidly at this dilution than at either 3 or 12% solids. For example, the critical temperature of an 8% solids solution of resin A would rise from 9° to 16° in 2 weeks, while the critical temperature of a 12% one would rise from 7° to only 12° in the same time. If the resin did come down, further dilution with hot water was necessary. Resin A has been modified so that now both A and B will not coagulate above 0°C. unless the materials are too old.

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* The per cent resin solids mentioned in this paper have been determined by the W.C.A.M. 2.1 method.

Occasionally, a barrel could not be used in proper rotation. The best method of checking its dilutability and usefulness was to find the critical solution temperature of a 3% solids solution. The sample should be diluted with distilled water and cooled with ice and coarse salt. If the temperature at which a white gelatinous precipitate forms is above 1 °C., care must be taken in using this resin. By the way, the reason for making the test at this low concentration is that a definite precipitation point exists. At higher solids, there is a considerable viscosity change but a precipitate may not be formed.

Before passing from the subject of handling to that of comparing urea-formaldehyde resins, it should be noted that a new system was installed during 1956–1958. The company now has two stainless steel pumps driven by variable speed motors and drawing from two 17,500-lb. capacity vinyl plastic storage tanks. These tanks are filled by an unloading pump which will also discharge resin from storage into barrels. In addition, the tanks are translucent so that inventories can be taken without a gage. This system has many more advantages.

Very briefly, they are: elimination of the dilution stage; positive proportioning by a pump instead of a rotameter; no lost paper due to rotameters sticking; two pumps instead of one, making the paper machines independent; lower resin costs with the elimination of drum shipments; and cleaner resin. With these accurate pumps, it is possible to make a comparison of the two cationic urea-formaldehyde compounds.

RESIN EVALUATION

Many factors beside the application rate are important in choosing a resin. To make a proper evaluation, it is necessary to have the averages of the following tests; brightness, absorbency, dry and wet tensiles, the headbox and finished paper pH, and also the extent of drier adhesion.

Considerable care is required to select the compound best suited for the grade conditions. For example, current trials indicate that resin B gives less Yankee drier coating than A on a butcherwrap paper having a furnish of semibleached kraft. When using resin B, the machine has very few breaks or doctor changes, but the exact reason for this effect is obscured by two facts.

First, the Yankee temperature is near the plastization point of both resins and this point is different for each material. Second, pitch development may be an important factor. Analysis of the drier coating indicates the presence of pitch and up to 5% of resin A.† The pitch-resin combination has a low plastization temperature and is tacky enough to stick the sheet quite firmly to the drier. With a change in conditions, this might also happen with resin B.

Passing to the physical tests, the brightness readings should be taken on off-machine paper and on samples which have been stored in a conditioning room for 2 and 12 weeks. These intervals were chosen because they are close to the minimum and average product life.

In comparing the effect of resins on brightness, several facts must be kept in mind. First, it has been observed that large amounts of acid, and to a lesser extent, alum, will cause a decrease in the 2 and 12 week values. This is most noticeable on fully bleached furnishes but shows up in towelling as a yellowish discoloration accompanied by a small change in the photovoltmeter readings. High resin feeds will also bring about a marked decrease in the 2 and 12-week values but with only a slight change in the initial brightness. A typical pattern for the combined effects on a bleached groundwood and semibleached kraft furnish are: off-machine paper brightness with acidification but with no wet strength—66.0 photovoltmeter units; off-machine paper with acidification and resin applied—64.0 units; paper 2 weeks old—63.0 units; 12 weeks old—62.0. On bleached kraft and bleached groundwood furnishes, the respective readings would be approximately 71.0, 69.0, 68.0, and 67.0 Photovolt units for a normal overall loss of 4 points in 12 weeks. In conclusion, machine trials indicate that resin B will give roughly 1 point extra brightness at all test intervals and so it has been chosen for an associated grade.

The next test to be considered is that of absorbency. At Westminster, the time in seconds for 0.1 ml. of distilled water to be completely absorbed is taken as a measure of this property. The readings do not change significantly with either resin on towel stock.

The most important factors appear to be the refining and per cent stretch in the finished paper. For example, with high refining, 0.10% rewetting agent and 0.5% alum, absorbencies of 50 sec. initially and up to 75 and 125 sec. in 2 and 12 weeks, respectively, were not unusual. By eliminating the alum, these figures were reduced to the 40, 55, and 75-sec. level for the three test intervals. With a small change in crepe to improve the softness, the absorbency values dropped to 30, 45, and 60 sec., respectively. As a final step in improving the softness, the refining was reduced and then, even with 1.0% alum, the values were roughly 15, 20, and 20 sec. for the three test periods. It appears that alum will only size the paper when the stock is refined. Lastly, changes in the drying conditions will alter the initial absorbency considerably while the 12-week values are apparently dependent on the drier temperatures.

WET-STRENGTHENING EFFECT

Finally, to study the wet-strengthening effect itself, the following minimum data should be recorded; the off-machine wet and dry tensiles, wet tensiles on 2 and 12-week old conditioned samples, and also wet tensiles on artificially cured paper at the time of manufacture to give a control value.

There will be some variance in the dry tensile values, depending on the degree of cure. Wet strength agents generally increase the initial dry strength from 1.0 to 5% of the untreated paper values. Up to 15% gain in the dry strength may be obtained after 12 weeks' storage.

The following test values illustrate toweling wet strength development: initial dry strength—75 oz.; off-machine wet tensiles—6 to 8 oz.; artificial cure value—30 oz.; 2-week natural aged—22 oz.; and a 12-week final test of 27 oz. Notice the rapid cure as indicated by the uncured and 2-week wet tensiles.

A graph of the above readings is often helpful. The line joining the point for the initial dry strength with that for the 12-week wet-tensile reading is probably the most important. Provided a number of conditions have been observed, the slope of this line is a good indication of resin efficiency.

The most important of these conditions are that the paper have uniform physical characteristics and exhibit uniform pH. The following observations have been made in studying resin efficiency. First, the acidity of the paper at the Yankee drier is the critical value. The headbox pH is a good control value, provided the paper does not show pH reversion, which may occur if poorly washed pulps have been furnished. Second, some alum in the furnish appears to give more rapid and consistent cures in the range of 4.0 to 4.5 units. If acid only is used for control, there is more variation in all the tensile values, particularly the 12-week ones. The best results are obtained when the pH is below 4.2 units. Third, as the pH is increased, particularly above 5.0 units, the deviation of the acidity readings must be considered. It appears that the results are comparable for papers made in the 4.2 to 5.0 range when the average values agree within 0.2 unit and when the variation from these averages is less than 0.20 unit. Above 5.0 units, it is very difficult to make any statement regarding efficiency as many other factors beside the acidity become important.

† A Kjeldahl nitrogen determination was used to estimate the per cent resin.

One final point—if the graphs described previously are used to find the most efficient resin, then the pH of the made paper at the various test intervals should be checked for reversion. In addition, the initial off-machine wet tensiles should be about the same percentage of the dry strength for each resin. If the cure rate varies considerably, the initial dry strength may be affected.

FURNISH CONSIDERATIONS

Before describing some of the problems in papermaking, the furnish must be considered. In general, the resins seem most effective on unbleached stock and more efficient on kraft rather than sulfite pulps. Addition of wet strength agents increases the tendency of the fibers to agglomerate. In extreme cases, the sheet will be quite blotchy or foam may build up. Changes in pulp types and proportions has not been a major problem except when a poorly washed pulp was used. Excessive foaming occurred frequently when using semibleached krafts, probably due to the bleach salts left in the pulp. pH measurements were taken on all shipments and those having a high reading were most likely to give trouble. This tendency to foam when the initial alkalinity is high applies to the groundwood produced at the Westminster Division also, despite acidification of the stock at the hydrapulper.

It has been found satisfactory to furnish all chemicals, except the wet strengtheners, at the hydrapulper. The order of addition is: tinting dye, broke, groundwood, chemical pulp, wetting agents, if any, and last, sulfuric acid and alum. Approximately 0.10% wetting agent was used until a year ago. The refining was reduced for more softness and the absorbency improved so much that no agents are required.

The amounts of alum and acid used have varied depending on the company's objectives. Five years ago, the chemical costs were quite significant and therefore a combination of 0.10 to 0.20% H_2SO_4 and 0.20 to 0.60% alum was used with high refining to get maximum wet and dry strengths. The alum caused poor absorbencies but helped wet-strength development. By switching over to acid only, the absorbency was considerably improved. Not until the refining was drastically reduced and the crepe increased was it possible to drop the wetting agent and use alum alone without impairing the absorbency.

Before furnishing a new chemical, it is very important to check its compatability with those already in use. For example, the original cationic wetting agent was quite stable to concentrated acids and the other towel additives. The machine crews found this compound was a good defoamer on other grades too. It seemed strange, then, that foaming increased if extra reagent was added, particularly if there was cooked broke in the furnish. It was observed that the foam was most severe if broke from a sized grade was used. In the laboratory, it was found that this compound would give large volumes of stable foam with rosin size. It is fortunate that this compound was not used as a defoamer on highly sized grades.

IMPORTANCE OF REFINING

The next step in the process to be discussed is that of refining. The resin polymer must be added after beating to get efficient results. The wet-strength polymers appear to have a wet bond that is weaker, or at least less mobile, than the rosin size fixation. Current data suggests that rosin sizing may be decreased up to 20% by adding all chemicals before refining. Past experience indicates that approximately 40% loss in wet-strength efficiency would occur under the same conditions if all the wet strength additives were beater loaded.

To determine the effect of refining, a urea-formaldehyde polymer was added at the Hydrapulper. The subsequent beating with a jordan and two Mordens reduced the wet tensiles approximately 50%. Another time, the resin was pumped in just before the jordan, causing a reduction in wet tensile of 15 to 20%.

Changes in refining influence wet strength results even when the resin is added after the stockmaking equipment. For example, in November, 1954, towels were made using a Morden and a jordan. The following month, another Morden was put into operation and the per cent wet-strength‡ rose from 24 to over 30 without any apparent change in the other conditions. The above results indicate that fibrillation plays an important part in making the fiber-resin bond effective. On the basis of several observations, it appears that mordening is more significant than jordaning. One logical guess at the reason is that the wet strength may be rendered less effective by attachment to the short fibers created by the jordan; these fibers may not contribute to the overall paper strength as much as the long ones and so resin bonded to these fibers may be, in effect, partly wasted. Before considering the next topic, it was observed that the hollander beaters appeared to give better results than the mordens. Unfortunately, it was not possible to get an estimate of the power actually spent on beating the stock.

POINT OF ADDITION

The last section of this essay describes the problems connected with the paper machine starting with the point of resin addition. Melamine and the urea-formaldehydes are usually added to the fan pump makeup box on the towel machine. The addition point is varied to suit the machine conditions. For example, a few years ago, some foaming occurred on every run of a related grade. It was found that a vortex was formed in the fan pump makeup box which led to air entrainment. With the resin added at this point, there was only 40 sec. mixing time before the stock was laid on the wire. It was almost impossible to keep the foam down by chemical means. The problem was solved by feeding the wet strength into the thick stock after the jordan rather than into the white water at the fan pump box.

Since water plays such an important role, the white water system could be considered as another unit in the manufacturing process. The points to be discussed next, concern resin retention, pH control and water supply, drainage effects, and slime control.

POLYMER RETENTION AND MIXING

Adding the wet-strength agents to the white water is a severe test of their retention properties. One supplier has shown that generally two-thirds of the polymer applied is retained in the paper. Undoubtedly, some of the resin is lost by absorbtion on the "flour" which passes through the save-alls. Another portion may be lost due to the variations in the polymer chain length and the receptivity of the pulp. Finally, one supplier has shown that the retention time is extremely short so that good mixing is the factor to be watched.

The mixing and retention times were checked using a basic dye, methylene blue, as the tracer. This cationic compound was selected because of its very good retention characteristics. It was estimated that 20 sec. are required for the stock to pass from the regulator to the fan pump, another 40 sec. was required for the stock to reach the slice, and a fraction of a second to reach the Yankee drier.

It was noted that there were slight variations in tint across the sheet. This was due not to poor mixing but to uneven weight distribution. When the dye solution was added at the fan pump makeup box, the water was colored as well as the stock. If the dye was added at the regulator, the paper at the Yankee was tinted but no color was observed in the fan pump water. This fact led to two further trials.

First, the point of resin addition was changed from the white water to the after jordan breather pipe. There was a

‡ In this case, per cent wet-strength = (12 week wet tensile/initial dry tensile) × 100.

slight increase in the wet tensile results. This improvement might have been due to better mixing and longer retention or, more likely, greater retention on the long fibers.

Second, a fixation agent was tried on the bleached kraft-bleached groundwood furnish as well as the semibleached kraft-bleached groundwood mixture. Because of the similarity between the fiber bonding shown by direct dyes and the U. F. polymers, a polyamine direct dye-fixing agent was selected for the trials. A slight increase in wet tensile was observed, particularly on the bleached kraft furnish. In both cases, the overall cost was increased. These compounds are not economical unless the resin usage is quite high or unless a large proportion of bleached sulfite is used.

pH CONTROL AND WATER SUPPLY

To get maximum efficiency, the pH of the paper at the Yankee must be carefully regulated. If the reading is over 6.0 units, poor development will result; if the readings are below 4.0 units, degradation of the paper can take place. In most mills, acidity measurements are not taken on the off-machine paper. However, it is wise to take periodic readings if pulps with a high initial alkalinity are used. There is a possibility that pH reversion will take place during storage.

For control purposes, the pH was originally determined on a sample drawn from the headbox. The readings were quite erratic due to the large volume of alkaline fresh water added at the shower tank when the savealls were stopped. The sampling point was changed to the line leading from the white water pit to the fan pump to give values reflecting only the major changes in pH.

With the installation of an automatic pH recorder and improved white water controls, the sampling point was changed to the discharge side of the fan pump itself. The values at this point, in the headbox, from the suction boxes and on the wet Yankee broke are all in close agreement. This has resulted in much more uniform results off the machine and at the consumer level. Only when the machine shuts down does the acidity become erratic. The addition of dissolved alum is the safest and quickest way to correct for high pH after a shutdown.

Since large volumes of water are required for papermaking, the quality of the water supply greatly influences pH control. Experiments indicate that roughly two-thirds of the acid used is required to offset the alkalinity of the fresh water. With the more alkaline pulps used two or three years ago, about half the acid was consumed in neutralizing the fresh water. There is a seasonal variation in the quantity of acidification agents required. During the freshest period, it is not unusual to double the amounts of sulfuric acid and alum.

COLLOIDAL CONSIDERATIONS

With normal resin feeds of 1.0 to 1.5% resin solids, the concentration of the active material is about 25 to 75 p.p.m. It is quite likely that small amounts of foreign matter introduced with the fresh water as well as the pulps will influence the wet strength results. Indeed, several mills have noticed a seasonal variation in U. F. resin efficiency. There is an obvious need for a detailed investigation of the colloidal systems existing in resin-treated white waters.

When these colloidal systems are upset, the results at the wet end are sometimes spectacular. For example, a few years ago a quick change was made from a U. F. resin to the melamine polymer, causing the water to carry over the suction boxes and into the couch. The magnitude of this effect was badly underestimated by the laboratory. Several freeness tests had been taken using 0.30% suspensions of after-refiner stock at 20°C. The results indicated that melamine reduced the freeness 3 points while the urea-formaldehydes increased the readings 3 points. Apparently this test does not properly reflect the considerable differences in drainage rates.

Finally, addition of wet-strength agents to the white water often brings on the formation of a colloidal slime. This is particularly noticeable when large quantities of alum are furnished and when alkaline pulps are neutralized with alum only. At the Westminster Division, chlorination of the white water is the routine slime control agent. However, the U. F. resins prevent the formation of a free chlorine residual so that a low-toxicity compound must be furnished. Tri-butyl tin oxide, Busan, and V-10 have given good control.

BROKE TREATMENT

Broke handling is the last topic of this essay. Originally, all broke up to 8 hr. old was furnished without special treatment. If the off-machine cure was below 6-oz. for a sheet having an artificial cure of 32 oz., then the broke could be beater loaded without cooking. For control purposes, the off-machine wet tensile was taken twice a shift to catch any changes in the cure rate. Increasing resin feeds coupled with the use of alum has forced the beater room to cook even the machine trim to prevent stock spots. Other factors which will give high initial cure are; low pH, overdrying the paper, higher Yankee temperatures, increased refining, or the use of melamine resin.

Paper not suited for direct furnishing plus the converting broke is cooked in a breaker beater using steam and sulfuric acid to hasten the repulping. The stock is heated to 180 to 200°F. at a pH of 4 to 5 units. If the slushed broke is not immediately discharged, up to 12 points loss in brightness may occur due to the acid attacking the mild steel beater walls. The use of a phosphate sequestering agent reduces the loss to approximately 6 points. The use of city water instead of filtered river water will give another 1 to 2 points recovery in brightness, so that the overall loss due to cooking is generally about 4 Photovolt meter units.

It is often necessary to know whether or not a paper contains wet strength when the broke is mixed. If a natural wet tensile does not give an indication, the following test solutions may be used. A drop of 1% phynylhydrazine hydrochloride in 40% H_2SO_4 is placed on the paper. After 30 sec., a drop of 10% aqueous ferric chloride is placed on the spot. An instant red color indicates presence of a U. F. resin, while a slowly developing pink color indicates the presence of melamine formaldehyde. This method was described in the "Canadian Pulp and Paper Association's Condensed Translations" for July, 1955.

CONCLUSION

In conclusion, the most striking fact is the number of problems encountered in applying just six chemicals. This fact emphasized the need for careful laboratory evaluation of changes in the chemical furnish and the need for an investigation of the colloidal conditions existing in resin-treated white waters.

The writer wishes to thank the management of the Westminster Paper Co. for permission to submit this essay to the Shibley Award Committee.

A Shibley Award paper presented at the meeting of the Pacific Section of TAPPI, held in Camas, Wash., April 7, 1959.

Fifteenth

Engineering Conference

Robert Meyer Hotel

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Oct. 24–28, 1960

Design and Operation of a Pilot Plant Coater

ERIC D. JONES and NORMAN H. LAUGHREY

Current interest in coating machinery indicates a trend wherein extreme versatility is desired in order to handle a wide range of coating materials and webs. This paper discusses the reasons for research and development in coating and its importance in relation to the proper selection of equipment for commercial production. From the results of pilot plant coating studies are gathered the data to insure the proper equipment and the features and versatility required in production machinery. Also discussed is the mechanical design of the equipment and the features built into it to provide maximum flexibility for research and development work. Various methods of coating are described including the air doctor, blade coater, and gate roll coater.

KEEN competition during recent years has forced the paperboard industry into an all-out drive to diversify and improve its products. Manufacturers have been especially active in developing new coating applications to improve the protective properties and the printing characteristics of folding carton stock. These efforts are already producing spectacular gains in coated paperboard quality. Further improvements are in store for the immediate future, and radically new developments may be closer at hand than most of us realize.

Experimental coating work is being carried out in the paperboard industry on pilot coating machines of various sizes and of many different designs; however, all have several common advantages. First of all, a pilot coater provides the manufacturer with a means for evaluating coating materials and for studying operational variables in the laboratory rather than in the mill where machine time is too valuable to allow prolonged experimentation. If the pilot equipment is sufficiently adaptable, different coating processes can be evaluated and altogether new procedures investigated.

General coating principles may transfer readily from one field of coating to another, but unfortunately the specific techniques that work well in one operation may not be applicable in another operation if the base sheet and end-use requirements differ appreciably. Crossett Paper Mills has a 210-in. cylinder machine producing a variety of grades of bleached foodboard. The printability requirements of some of the grades manufactured made the addition of coating equipment highly desirable if not an absolute necessity. The problem of selecting a suitable coating method was assigned to the Research Division of The Crossett Co. Because cylinder board has properties which are decidedly different from the properties of fourdrinier board, it became obvious that Crossett would have problems which are peculiar to cylinder machine operation and cylinder machine products. The numerous problems involved finally led to a decision to enter the coating field on the pilot plant level.

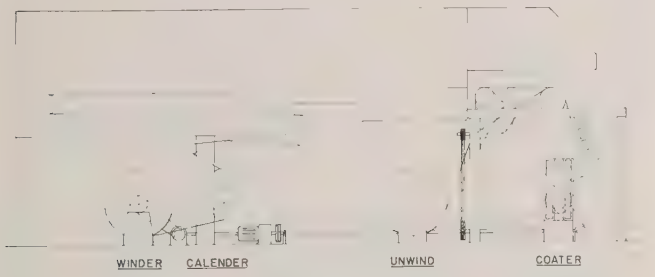

Fig. 1. Return pass drier

ERIC D. JONES, Research Division, The Crossett Co., Crossett, Ark., and NORMAN H. LAUGHREY, Dilts Division, The Black-Clawson Co., Fulton, N. Y.

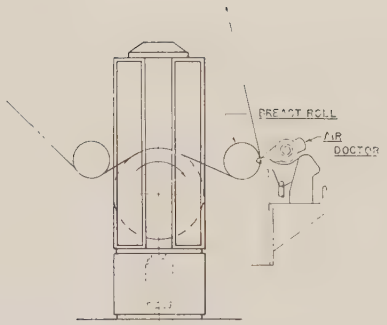

Fig. 2. Air doctor

The Dilts Division of The Black-Clawson Co. and the Research Division of The Crossett Co. based the design of Crossett's pilot coater upon the following general specifications and considerations:

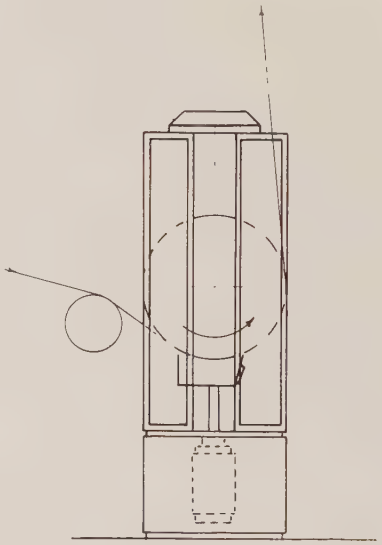

Fig. 3. Flexiblade coater

1. The air doctor is used successfully in many installations to apply both protective and pigmented coatings. Its versatility makes the air doctor a desirable part of the pilot installation.
2. Because its leveling action provides an excellent printing surface, a blade coater should be a part of the installation even though streaking and coat-weight limitations are known disadvantages.

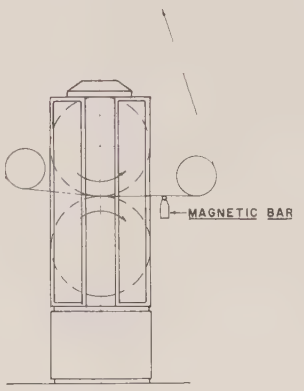

Fig. 4. Magnetic metering bar

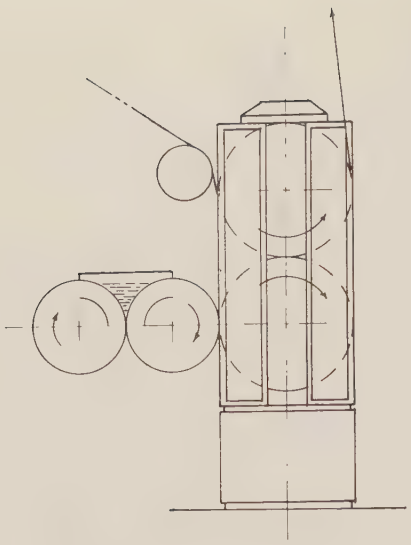

Fig. 5. Gate roll coater

3. A size press should be included because it is widely used as a pre-treatment in coating.

4. The basic frame should be readily adaptable to additional coating methods such as roll and gravure coating.

5. Changeover from one coating method to another must be accomplished with comparative ease and without requiring a great deal of time and effort.

6. The speed of the coater should be variable and should cover the range of operating speeds of the cylinder machine.

7. The coater should trim up to 36 in. so that paperboard converters can evaluate experimentally coated samples in full scale converting operations.

8. The drier system should be versatile so that different drying processes can be studied.

9. All electrical equipment should be made explosion-proof to permit studies of methods for applying functional coats from solvent as well as water systems.

The coating line is shown schematically in Fig. 1. The line shaft drive is powered by a 30-hp., a.c. motor. A magnetic clutch coupling between the motor and line shaft allows speed control in the range of 50 to 500 f.p.m. A right angle takeoff of either clockwise or counter-clockwise rotation drives the bottom coater roll through a variable PIV. Another takeoff feeds a gear train which controls the speed of the drum driers, the slat conveyor in the conventional drier, and the top coater roll. A third takeoff through a gear box drives the

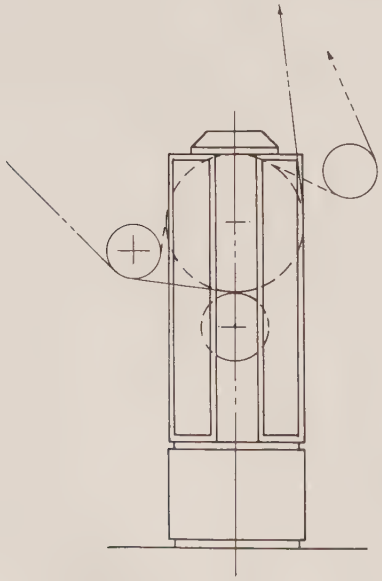

Fig. 6. Gravure coater

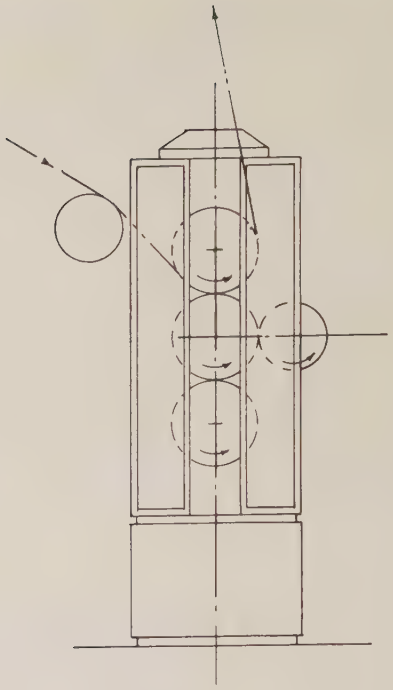

Fig. 7. Contracoater; reverse roll coater

center roll in the calender stack and also provides power to the winder through an air-loaded slipclutch.

The drying section incorporates a gas-fired infrared unit, two drum driers, and a 45 ft. double pass convection-type drier. The upper zone of the oven is 30 ft. long, the lower zone 15 ft. long. In these two zones temperature is controlled by the amount of steam flowing through heat exchangers. As an additional precaution in making the installation explosion-proof, an exhaust hood was built into the drier system to remove solvent vapors from the oven. The drier section is designed so that high velocity, high-temperature air drying can be added in the future.

Following the drier section, the web passes through a three-roll calender stack, having the upper and lower rolls air-loaded to a maximum load of 160 lb. per linear inch, to a single position rewind stand driven from the line shaft through an air-loaded clutch. The web lead shown in Fig. 1 is for the vertical size press adaptation.

The basic frame of the coating head can easily be adapted to the air-doctor operation, as shown in Fig. 2. In this operation the color roll and color pan are raised into operating position by means of the two air cylinders at the bottom of the frame. The slow turning color roll applies an excess amount of coating to the sheet and the excess is removed by the air doctor.

Another variation in the Flexiblade adaptation is shown

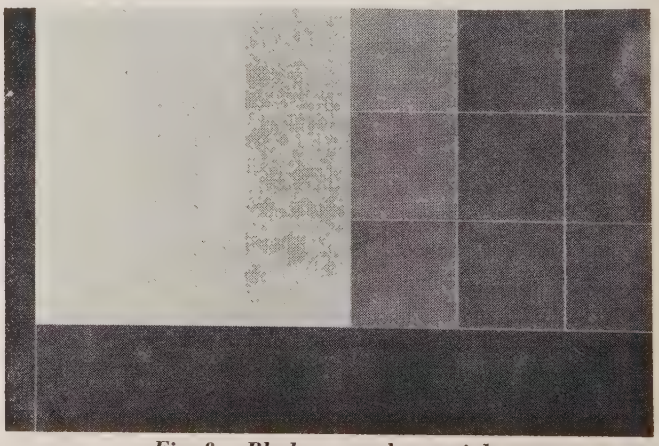

Fig. 8. Blade coat; low weight

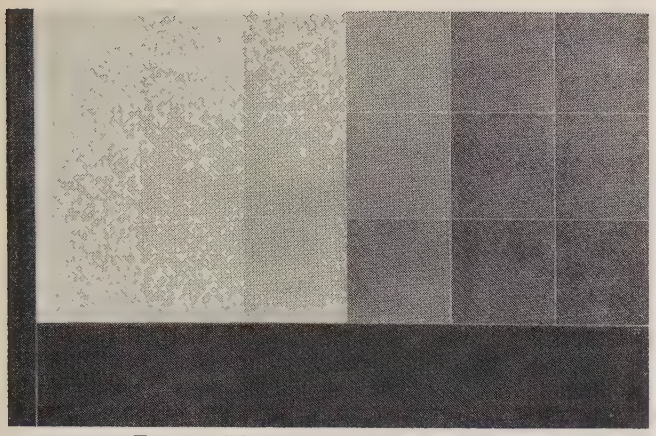

Fig. 9. Blade coat; normal weight

in Fig. 3. In this operation the rubber-covered backing roll is locked into place in the frame and the coater is raised into operating position by the air-loaded cylinders. The amount of coating applied may be controlled by varying the pressure in the coater head and by changing the pressure and angle of the blade.

Another type of coating setup can be built by the use of magnetic metering bar coater in Fig. 4. The bar itself is generally $^1/_4$-in. stock supported in a V notch, which not only cradles it rigidly but acts as a doctor to the bar as it rotates against the web travel. This coater is used primarily as a pre-coater in the paperboard industry.

The gate-roll coater shown in Fig. 5 can be easily adapted to the machine by adding the two metering rolls.

Offset or gravure type coating is shown in Fig. 6. In this coater the depth of the etch controls the amount of coating applied. In direct gravure the web contracts the etched roll directly; in offset the coating is transferred to a second roll and thus to the paper.

Also considered for future application is the four roll reverse roll coater in Fig. 7. Reverse-roll coating may be used for any type of coating although it is probably best adapted to solvent systems. With this coater it is possible to control film thickness within close limits.

The following coating procedures, which were evaluated in preliminary coating studies, demonstrate the versatility of the basic coating machine:

1. Single- and double-blade coats.
2. Single- and double air-doctor coats.
3. Blade coat covered by air-doctor coat.
4. Air-doctor coat covered by blade coat.
5. Above combinations with size press pre-treatment.

Paperboard used in coating experiments is made on Crossett's cylinder machine with only the wet stack lowered. The use of 40-in. diameter rolls, all taken from the same reel, minimizes the paper variables and makes it possible to duplicate results with any given procedure and to compare results from different procedures. The advantages of being able to perform several experiments on one set of paper are obvious.

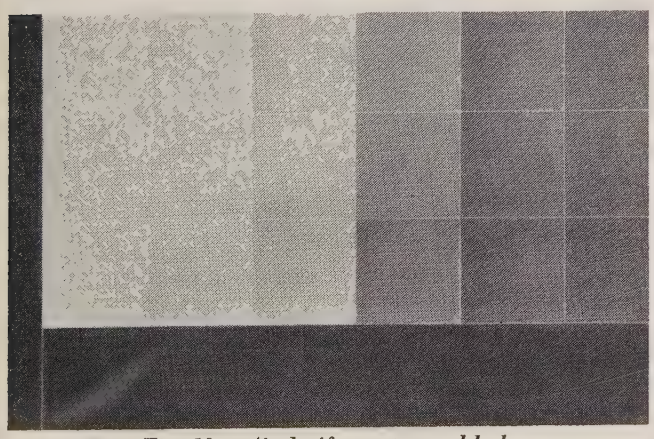

Fig. 10. Air-knife coat over blade

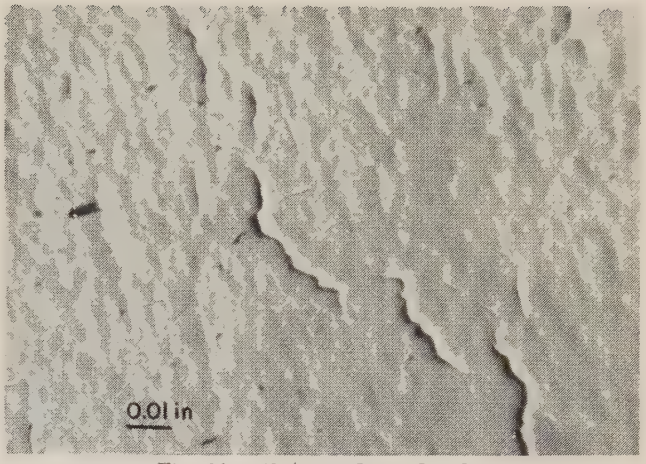

Fig. 11. 50% synthetic binder

The effects of varying the coat weight applied by a blade coater on printability of cylinder board are shown in Figs. 8 and 9. The print mottle on low weight coats (Fig. 8) is unacceptable but is much improved at a higher pickup (Fig. 9). A top coat applied by the air doctor over the single-blade coated sheet results in a good printing surface (Fig. 10). Since all three of these samples came from the same roll of paper, the paper variable was eliminated and all differences are caused by coating process variables.

Preliminary studies on the effect of rate of drying have been made with the coater. These studies have uncovered problems which seem to be associated with double coating. The photomicrographs in Figs. 11–13 show the effects of substituting increasing amounts of a synthetic resin binder for starch and protein binders in a clay coating. In this study the base sheet was first coated with a blade coat and then covered by different air-doctor coats. Machine speed, drying temperature, and base sheet were identical for all three samples.

A particularly useful feature on this machine is the calender stack. By repeated passes through the calender stack the effect of calendering on printability and other sheet properties can be determined.

An intangible benefit from a pilot coater is training of personnel. In addition to learning how to deal with many of the problems associated with coating paperboard, it is necessary for coating personnel to become acquainted with product evaluation and printing techniques.

The field of functional or protective coating presents an even greater challenge to paper manufacturers than does the problem of improving printability. This pilot machine

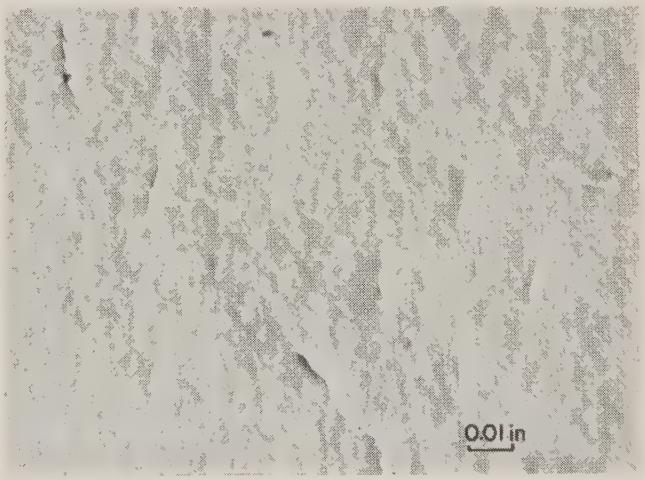

Fig. 12. 70% Synthetic binder

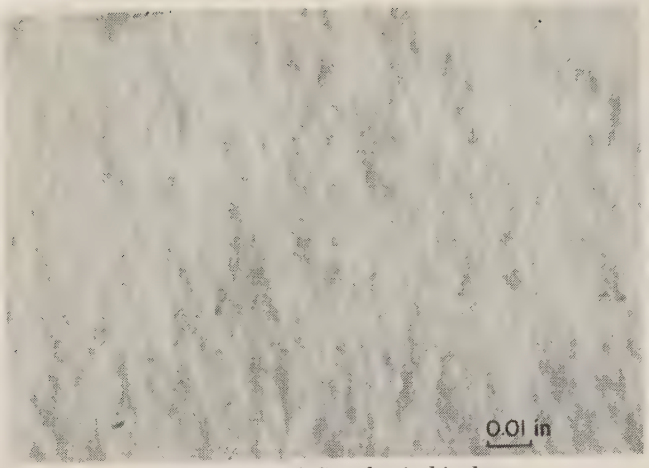

Fig. 13. 100% Synthetic binder

is well suited to carry out the research needed to study methods of application as well as the properties of different film formers used in protective packaging.

In addition to providing an opportunity for the development of new and better paper products, the experimental coater provides additional tools to improve the range and quality of technical service to paper and paperboard converters.

Received June 6, 1960. Presented at the 11th Coating Conference of the Technical Association of the Pulp and Paper Industry, held in Chicago, Ill., May 23–25, 1960.

Improving Black Liquor Oxidation Efficiency of Packed Towers

WILLIAM B. WEST

A commercial scale oxidation system (two towers) was surveyed in an attempt to improve the overall oxidation efficiency of the towers. Data obtained indicate that significant improvements in over-all oxidation efficiency can be realized by operating multiple towers in series rather than in parallel flow. The results indicate that the capacity coefficient for a given tower packing increases with increasing liquor flow rates and that the capacity coefficient for complete oxidation approaches the maximum capacity coefficient for a given packing as the mass flow rate approaches infinity.

The oxidation or stabilization of sulfur compounds in black liquor from the kraft pulping process has long been recognized as a worth-while development and has found application in many mills, particularly in areas where a pressing odor problem exists.

While the reduction of air polution is one of the primary advantages offered by black liquor oxidation, some of the other advantages are listed as (*1*):

1. Maintenance of the sulfidity of the cooking liquor at a higher level than is ordinarily obtainable with straight salt cake makeup.
2. Reduction of the amount of lime required for causticizing because of higher sulfidity.
3. Reduction of corrosion in vacuum and direct contact evaporators.

The oxidation of black liquor is theoretically a relatively simple process which involves bringing air into intimate contact with black liquor from the washing stage. This process is generally carried out in packed towers in which liquor flows down through the tower in contact with the air required for the oxidation reaction.

The primary reaction which takes place during oxidation is the conversion of sodium sulfide (Na_2S) to sodium thiosulfate. The elimination or reduction of sulfide ion in the black liquor has been found to reduce or eliminate the release of hydrogen sulfide gas from the vacuum and direct contact evaporators due to the conversion of the sulfide to the more stable thiosulfate. This results in the increased retention of sulfur in the system and a reduced discharge of malodorous sulfur compounds to the atmosphere.

The Central Research Department of Crown Zellerbach Corp. was requested to survey the oxidation system at Crown Canada's 500 ton per day kraft mill at Elk Falls, B. C., and make recommendations for improving the overall oxidation efficiency of the system. Two oxidation towers were in operation primarily as an odor control measure. Tests run by the Elk Falls mill had indicated that the oxidation efficiencies of the towers were generally too low to be of much value for odor control purposes.

The oxidation towers were of a type developed in Canada by the British Columbia Research Council and were packed with asbestos cement plates with a diamond imprint on the major surfaces designed to create turbulence for mass transfer in the liquor as it flowed down over the plate surfaces. Each tower had a packed height of 34 ft., a cross-sectional area of 33.4 sq. ft., and contained 24,000 sq. ft. of packing surface area. The packing plates were mounted vertically on 1-in. centers.

The liquor flow from the washers was split equally at the towers where it was evenly distributed over the packing surfaces at the top of the tower. If flowed down through the towers in parallel flow to a sump and was pumped to storage.

Air for tower operation was drawn through an intake duct and introduced at the top of the towers to flow concurrently with the liquor down through the tower, and discharged back up the tower through a vertical central duct to the atmosphere.

In addition to the air required for tower operation, blow and relief gases were introduced to the towers through the same ducting. The reason for this procedure was that various investigators had shown that some of the malodorous materials in the blow and relief gases were absorbed by the black liquor in passage through the towers (*1*).

An examination of the oxidation system as described indicated that three factors may have been contributing to the low efficiencies which were being experienced.

1. The towers may have been underdesigned, i.e., the packed volume may have been too small to sustain the mass transfer and air-liquor contact conditions necessary to complete the oxidation reaction.
2. The volume of air being supplied to the towers may have been inadequate to sustain the reaction.
3. The surges of blow and relief noncondensable gases being introduced with the air supply may have been diluting the air supply to a level too low for proper oxidation.

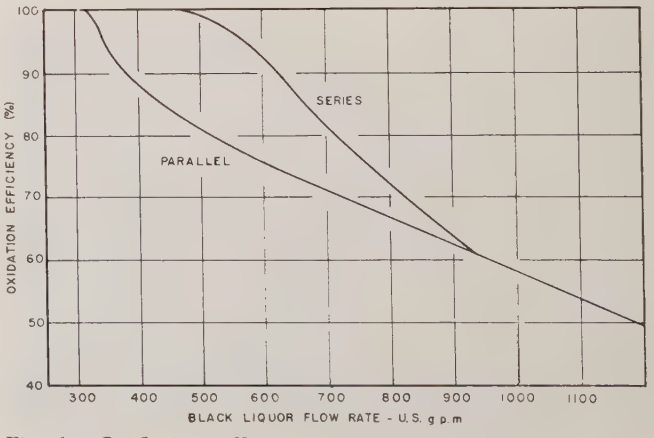

Fig. 1. Oxidation efficiency in series and parallel operation

William B. West, Associate Engineer, Central Research Dept., Crown Zellerbach Corp., Camas, Wash.

Table I. Capacity Coefficients and Tower Efficiencies at Various Mass Flow Rates

Prod. ADT/day	Flow rate, U. S. g.p.m.	Parallel tower operation, H = 34 ft. L, lb./hr./sq. ft.	Efficiency, %	A, lb./Na_2S/hr./cu. ft.	Series tower operation, H = 68 ft. L, lb./hr./sq. ft.	Efficiency, %	A, lb./Na_2S/hr./cu. ft.
188	300	2450	100	0.396	4,900	100	...
250	400	3260	87	0.460	6,520	100	...
290	465	...	...	...	7,440	100	0.600
310	500	3970	80	0.515	7,940	99	0.642
375	600	4900	76	0.602	9,800	90	0.713
435	700	5710	72	0.665	11,420	80	0.741
500	800	6550	68	0.720	13,100	72	0.765
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The system was surveyed with these three questions in mind. The liquor flow rate to the towers was varied over a wide range so that the tower efficiencies could be determined at different feed rates. Tower efficiencies were measured by determining the concentration of sodium sulfide in the feed and discharge liquor and are expressed as a percentage of the feed concentration. The potentiometric method of Barloe and Pascoe was used for the analysis of sodium sulfide.

Air volumes were determined through air flow measurements made in the intake ducting to the towers. The relative flows of blow and relief gases were determined in the same manner. Oxidation efficiency was determined both during and between blows to determine the effect of passing the blow gases through the towers.

The concentration of sodium sulfide in the tower feed was determined to be 6 g.p.l. or 5.5×10^{-3} lb. per lb. of liquor fed to the towers. This concentration remained fairly constant throughout the period of the trials, and could be expected to remain fairly constant as long as the cooking liquor sulfidity remains constant.

The air supply was indicated to be adequate to sustain the oxidation reaction based on the concentration of sodium sulfide determined. Other investigators have determined that an excess of about 300% theoretical oxygen based on conversion of sodium sulfide to sodium thiosulfate is necessary to sustain the reaction (*1*). In this case, the theoretical oxygen supply was from 500 to 600%.

The practice of venting the blow and relief gases through the towers was found to have no effect on the tower efficiency as tests made both during and between blows indicated the same efficiency for any given feed rate. The actual dilution of the air supply was about 10% during periods of blow and was not measurable during periods of relief.

The actual tower efficiencies at various flow rates are illustrated in Fig. 1 by plotting the equilibrium flow rate and corresponding pulp production rate against tower efficiencies. It can be readily seen that the efficiency drops off sharply as the production rate with its corresponding flow rate is increased. To achieve maximum efficiency and thus achieve the maximum odor control potential, the production rate would have to cut to 200 ton per day. The oxidation efficiency then drops off to about 66% as the production is increased to the maximum of 500 ton per day.

The average capacity coefficient for the towers can be calculated by the following formula (Table I):

$$A = \frac{L}{H}(C_I - C_D)$$

where

A = capacity coefficient, lb. Na_2S oxidized/hr./cu. of packed volume.
L = mass flow rate, lb. black liquor/hr./sq. ft. of cross-section area.
C_I = concentration of Na_2S in the feed liquor, lb./lb.
C_D = concentration of Na_2S in the discharge liquor, lb./lb.
H = packed tower height, ft.

When the capacity coefficient A is calculated in this manner, a significant increase in A is indicated with an increase in the mass flow rate L.

Analysis of these data indicates that an overall increase in tower efficiency could be achieved if the towers were operated in series flow rather than parallel. Series flow would result in the value of L (mass flow rate) being doubled for any given flow rate and if the relationship of increasing A with L holds, there should be an increase in the capacity coefficient A at any given level of tower efficiency.

Piping modifications were made which enabled the towers to be operated in series. The results are plotted in Fig. 1 and compared to the previous parallel flow curve. The results generally corroborate the theoretical advantage indicated for the series tower arrangement, with efficiency at any given flow rate higher than for parallel flow.

The capacity coefficients were calculated and tabulated in Table I. At any given level of tower efficiency, A is significantly higher for the series test, indicating that the increased mass transfer time or equivalent tower height obtained by series flow enabled the oxidation reaction to more fully approach completion at a given flow rate. This indicates that doubling the volumetric loading to the tower did not halve the retention time.

The value of the capacity coefficient reached the same maximum value ($A_{max.}$) for each flow arrangement indicating that this value is maximum for the tower packing under these black liquor conditions.

Based on the increase in A at the 100% efficiency level A_{100} at the point of inflection for series over parallel flow it can be concluded that as L is increased to infinity, the value of A_{100} would approach $A_{max.}$ for any given system. However, within the range of practical operating conditions, A_{100} is some value less than $A_{max.}$

The most economic tower design then, from the standpoint of packing efficiency would utilize as high a mass flow rate L and equivalent tower height as was practical so that A_{100} would approach $A_{max.}$

In terms of the Elk Falls system, the overall tower efficiency can be improved by operating the existing towers in series during periods when the pulp mill production is somewhat less than 400 tons per day, however, for the realization of the maximum odor control and recovery potential offered by oxidation, additional tower capacity is needed. The additional requirements can readily be calculated using the capacity equation since L and C are known for this system, and A for 100% oxidation can be estimated from the available data.

Several overall conclusions can be drawn from this work which should be of importance in the design of more efficient commercial towers and the evaluation of various packing materials.

1. The capacity coefficient increases significantly with the mass flow rate L and reaches a maximum for a given packing at some level of L.

2. The capacity coefficient for 100% oxidation approaches the maximum coefficient for a given packing as L approaches infinity. However, within the range of practical operating limits A_{100} is some value less than $A_{max.}$

3. The significance of equivalent tower height should not be overlooked as it appears to be a factor influencing overall tower efficiency.

Several factors may influence the value of the capacity coefficients for any given system, specifically the concentration of sodium sulfide and the type of packing used. However, the general relationships which were demonstrated by this study should hold for any system.

The results demonstrated by this work could be applied now to mills which operate similar multiple tower installations. While many such installations exist, it is doubtful whether any are presently operating their towers in series. Significant improvements in overall oxidation and recovery efficiency may be possible in many instances if existing towers are operated in series to increase the equivalent tower height.

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But we are proud that TITANOX titanium dioxide is being used to brighten the "rainy-day" bags that help the Times reach its subscribers in the most readable condition. It's this white titanium dioxide pigment that gives this waxed-paper stock the whiteness, brightness and opacity that makes the Times' advertising message stand out.

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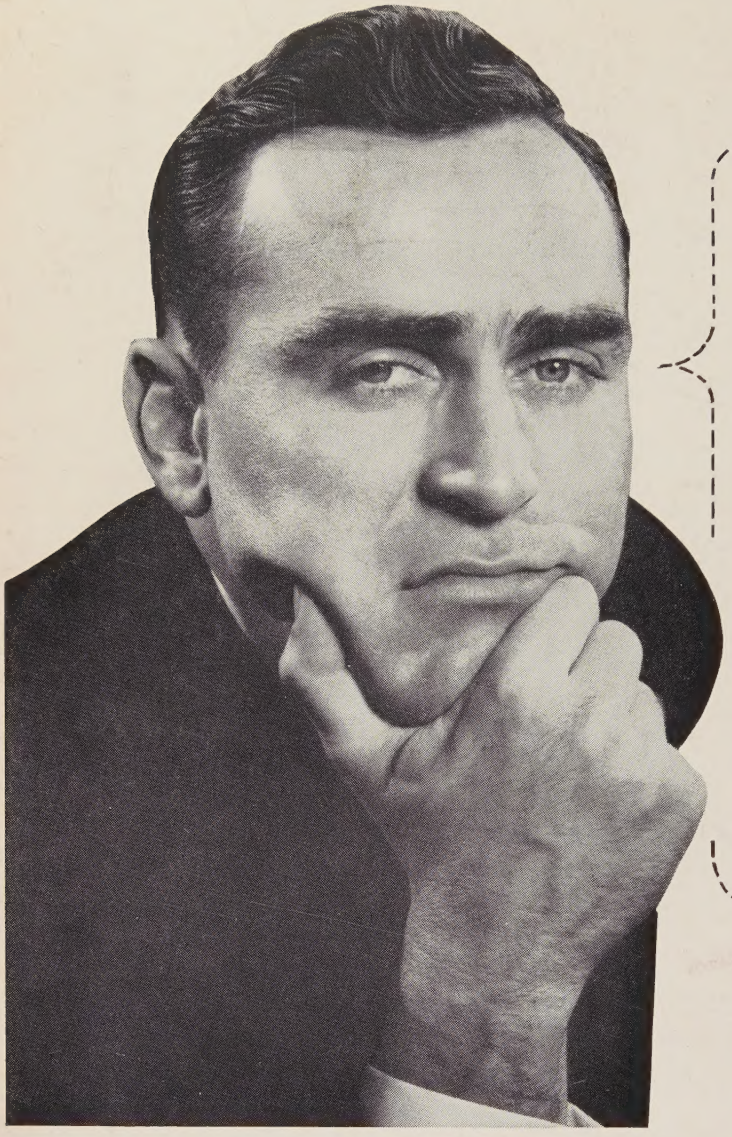
And as it is with paper, so it is with a wide range of other products...there is a TITANOX pigment for anything that needs whitening, brightening or opacifying. Titanium Pigment Corp., 111 Broadway, N.Y. 6, N.Y. In Canada: Canadian Titanium Pigments, Ltd., Montreal.

TITANIUM PIGMENT CORPORATION

SUBSIDIARY OF NATIONAL LEAD COMPANY

TITANOX

Thinking of coating or filling with clay?



Should we use a trailing blade... or an air knife?

Maybe it should be a roll coater... or a reverse roll?

What about a size press?

Double coating? Simultaneous or successive?

On or off the machine?

Wet or dry storage?

Fill at the secondary headbox?

Best place to furnish the clay?

There are plenty of tough decisions to make when it comes to coating or filling paper with clay. Using clay successfully involves a lot more than selecting a grade.

You'll find the Georgia Kaolin Company furnishes a lot more than just quality clay. The important extra is technical service. Georgia Kaolin has helped many companies, large and small, in solving problems of clay handling, clay make-down and coating-color formulation.

Georgia Kaolin technical assistance goes beyond ideas and opinions. The Sales Service Department operates pilot coating machines which can demonstrate the effects, on your own raw stock, of different formulations and various methods of coating. These facilities, plus Georgia Kaolin knowledge and skill, can be an all-important adjunct to your own research.

START BY CONTACTING US.

Georgia Kaolin Company 433 North Broad St., Elizabeth, N. J.

Fine clays from the world's largest kaolin plant